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WADC TECHNICAL REPORT 53-373

SUPPLEMENT 2

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A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM

MARY M. SOKAS, CAPT, USAF

MATERIALS LABORATORY

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FEBRUARY 1956

Statement A
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WADC TECHNICAL REPORT 53-373
SUPPLEMENT 2

A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM

MARY M. SOKAS, CAPT, USAF

MATERIALS LABORATORY

FEBRUARY 1956

**WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO**

FOREWORD

This report was prepared by Mary M. Sokas, Capt, USAF, Chief, Technical Data Section, Technical Services Branch, Materials Laboratory, Directorate of Research, Wright Air Development Center, Wright-Patterson Air Force Base.

The assistance of Mrs. Nellie Ragen, Mrs. Ruth Walden, Miss Helen Hines and Mrs. Sara Klenk in the preparation of this report is gratefully acknowledged.

Technical reports prepared by Materials Laboratory project engineers (those having no contract number) and Materials Laboratory contractors during the period 1 July 1954 - 30 June 1955 are abstracted herein. (Abstracts for a limited few reports were not available at this time but will be abstracted in the next Supplement.)

This report has been released to the office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. for sale to the general public. To order copies of this report, address inquiries to the Department of Commerce.

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The general public is invited to submit requests for copies of technical reports which are abstracted in this report to the Department of Commerce. Military organizations and prime contractors should write to ASTIA.

ABSTRACT

Two hundred and five (205) technical reports written during the period 1 July 1954 - 30 June 1955 are abstracted. These reports cover the following areas of research: adhesives, metallurgy, analysis and measurement, biochemistry, textiles, petroleum products, plastics, packaging, protective treatments and rubber.

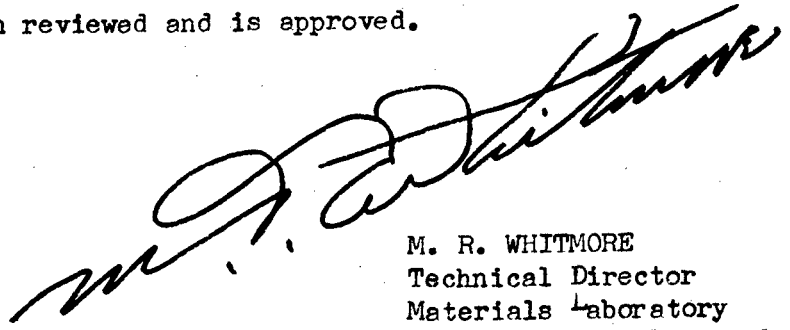
In Section II are listed ten (10) reports issued during July 1952 - June 1954 which were not mentioned previously.

As a final summary, a corrected numerical index of all the technical reports issued during the period March 1923 - June 1955 is provided.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

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ADHESIVES, STRUCTURAL

WADC TR 52-156 (Sup 1)

July 1954

SUBJECT: IMPROVED STRUCTURAL ADHESIVES FOR BONDING METALS
INVESTIGATOR: H. C. Engel
CONTRACT: AF 33(600)-23194
CONTRACTOR: Bloomingdale Rubber Company
ABSTRACT: The work of improving the structural metal-to-metal adhesive formulation that was previously developed under Contract AF 33(038)-21669 is presented. Shear strength test data of aluminum joints bonded with experimental modifications of the adhesive formulation and with experimental curing cycles and assembly procedures are listed. The strength data is the basis on which the conclusions are made as to the best formulation, its degree stability, and the optimum conditions for bonding which result in the best strength properties.

WADC TR 53-126 Pt. 2

August 1954

SUBJECT: ELEVATED TEMPERATURE-RESISTANT MODIFIED EPOXIDE
RESIN ADHESIVES FOR METALS
INVESTIGATOR: M. Naps
CONTRACT: AF 33(600)-6514
CONTRACTOR: Shell Development Company
ABSTRACT: A metal-to-metal adhesive which is useful at temperatures from -70°F to 500°F has been developed. The adhesive, designated as Formulation 422, is one package system based on a combination of one part of a solid poly-epoxide, EPON 1001, and two parts of a liquid phenolic resin, Plyophen 5023. The resins are co-cured with dicyandiamide at contact pressure and at elevated temperature (330°F). Aluminum dust is the reinforcing filler and a small amount of a copper chelate compound of 8-quinolinolate or diethylene-triamine is used to improve the thermal stability of the adhesive.

Bonds to aluminum from Formulation 422 have a shear strength retention of about 75% at 500°F; tensile shear strength is 2150-2500 psi at room temperature and 1645 to 1850 psi at 500°F. This shear strength is about 1000 psi after aging the unstressed bond for 70 to 90 hours at 500°F and 600 to 800 psi after 200 hours. The long time load strength at 500°F is about 800 psi. Shear strength retention at 500°F after cycling the unstressed bond between room temperature and 500°F is approximately 65% after 100 cycles.

Adhesive Formulation 422 is used as a pliable tape preferably with a glass fabric carrier. The storage life of the film is three to five weeks at room temperature and eight to twelve months under refrigeration (30 to 40°F).

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The adhesive is compounded by a hot melt process. Limited studies of a solvent process indicated that this technique may be more adaptable to large scale work, but offers no improvement in the room temperature storage stability and bond performance of the adhesive.

Formulation studies included evaluations of a large number of phenolic resins, reinforcing fillers, and heat stabilizers as well as a few curing agents other than dicyandiamide. Maximum adhesive strength was obtained, however, with the adhesive components given above. Two phenolic resins, Lebec 102210 and Conolon 1007, may be substituted for the Plyphen 5023 with some sacrifice (about 10 to 20%) in shear strength. Asbestos is the most promising reinforcing filler, other than aluminum powder. No other adequate curing agent for the adhesive was found.

WADC TR 53-294 (Sup 1)

April 1955

SUBJECT: DEVELOPMENT OF ROOM TEMPERATURE CURING STRUCTURAL ADHESIVES FOR METALS
INVESTIGATOR: Risto P. Lappala
CONTRACT: AF 33(616)-165
CONTRACTOR: Bjorksten Research Laboratories, Inc.
ABSTRACT: A previous investigation on room temperature curing metal-to-metal adhesives (WADC TR 53-294) resulted in the development of an acrylic adhesive which met the specified requirements except for its short pot life and open assembly time. This report describes a supplemental investigation which was made in an attempt to improve these properties.

A large number of formulations were prepared using acrylic monomers and carboxylic acids which had not been investigated previously. The best adhesive, on the basis of shear strength screening tests of aluminum-to-aluminum bonds, contained MPL monomer, methacrylic acid, Acryloid B-82, and glass fibers sized with vinyl trichlorosilane. This adhesive did not completely fulfill the requirements, but used alone or preferably in combination with the best adhesive of the previous investigation, it allowed 1/2 hour or more of open assembly time. The pot life could be adjusted by varying the proportions of catalysts.

A two-part formulation prepared late in the program was the only adhesive to meet the low temperature shear strength requirement. Time did not permit full evaluation of this formulation.

WADC TR 53-294

July 1954

SUBJECT: DEVELOPMENT OF ROOM-TEMPERATURE-CURING STRUCTURAL ADHESIVES FOR METALS
INVESTIGATOR: Johan Bjorksten, Risto P. Lappala, Luther L. Yaeger and Robert J. Roth

WADC TR 53-373 Sup 2

CONTRACT: AF 33(616)-165
CONTRACTOR: Bjorksten Research Laboratories, Inc.
ABSTRACT: The work described herein has been directed toward the development of a room-temperature low-pressure curing metal-to-metal adhesive suitable for the fabrication and field repair of certain airframe structural parts where the use of heating and pressurizing equipment would be impracticable or impossible. An adhesive designated as P-262A, consisting basically of methacrylic acid and methyl methacrylate, has been developed which meets most of the research objectives and possesses properties comparable to those required by Military Specification MIL-A-8331 (USAF). Its development, formulation, processing, and properties are presented. Further improvement lies basically in the lengthening of pot life and open assembly time toward which objectives further research and development work is contemplated.

WADC TR 53-477

September 1954

SUBJECT: SURFACE TREATMENT OF METALS FOR ADHESIVE BONDING
INVESTIGATOR: Robert S. Shane, Theodore L. Eriksson, Alexander Korczak and Dwight B. Conklin
CONTRACT: AF 33(616)-2055
CONTRACTOR: Wyandotte Chemicals Corporation
ABSTRACT: Preliminary studies have been made on the preparation of aluminum, stainless steel, and magnesium surfaces for adhesive bonding. The lap joint shear test at room temperature was used as the criterion. Tests on samples exposed to 95% relative humidity for 14 days and salt spray for 30 days are also included. A Dillon Universal Tester, as modified to provide motorization, pacing, and improved grips, is detailed. Bloomington FM-47 adhesive was used as the screening adhesive; other adhesives evaluated were Scotchweld No. 585 Tape, Shell Epon VIII adhesive, and Metlbond MN 3C tape. Eight other adhesives were examined in making a choice of screening adhesive. Approximately twenty different etchants were tried for stainless steel; a surface treatment is recommended as giving bond strengths substantially the same as those produced on aluminum using a modified Forest Products Laboratory surface treatment. In preliminary work on magnesium surfaces, eighteen etchant formulations were examined. Surface treatments AD and AE show mean shear strengths in excess of 2300 psi at room temperature without zinc chromate lacquer primer. A method for distinguishing between adhesive and cohesive failure is described.

WADC TR 54-98

August 1954

SUBJECT: INVESTIGATION AND DEVELOPMENT OF HIGH-TEMPERATURE STRUCTURAL ADHESIVES
INVESTIGATOR: H.N. Homeyer Jr., J.H. Preston and K.L. McHugh
CONTRACT: AF 33(616)-427
CONTRACTOR: Connecticut Hard Rubber Company
ABSTRACT: This report describes work on the investigation and development of structural adhesives from silicon materials for high temperature service conditions such as are encountered in certain aircraft operations. The results

WADC TR 53-373 Sup 3

of lapshear tests on aluminum-to-aluminum specimens bonded with a large number of silicone resins, rubbers, copolymers, and experimentally compounded mixtures are reported. The data presented shows that adhesives with considerable strength can be obtained and that the target objectives, although not yet reached, are not beyond the realm of possibility. It was found that shear strengths averaging over 2000 psi at 70°F, 700 to 900 psi at 300° F, and 400 psi at 500° F, can be obtained with unmodified resins.

WADC TR 54-153

December 1954

SUBJECT: EFFECT OF RATE OF LOADING ON SHEAR STRENGTH OF ADHESIVE-BONDED LAP JOINTS

INVESTIGATOR: R.E. Wittman

ABSTRACT: The effect of various rates of loading on the shear strengths of several metal to metal adhesives is presented. The loading rate ranged up to values as high as 10^8 psi per minute.

The shear strength for the more rigid adhesive types, vinyl-phenolic and epoxide, remained fairly constant at the increased rates of loading.

The shear strength of the less rigid types of adhesives, nitrile rubber-phenolic and nylon-neoprene rubber-phenolic, increased appreciably with increases in loading rate and at the highest rate (0.002 second to failure) the strengths were more than doubled, the maximum increase being about 230 percent.

Strengthwise, the more rigid adhesives had higher values when stressed statically (600-700 psi per minute) than did the less rigid types. As a group, these values averaged 4110 psi and 2960 psi respectively.

WADC TR 54-447 (Part 1)

February 1955

SUBJECT: WEATHERING OF ADHESIVE-BONDED LAP JOINTS OF CLAD ALUMINUM ALLOY

INVESTIGATOR: H.W. Eickner

CONTRACT: PO 33(038)-51-4326 E

COTRACTOR: Forest Products Laboratory

ABSTRACT: Lap-joint panels of clad 24S-T3 aluminum bonded with 5 metal-bonding adhesives were exposed to weathering at the Panama Canal Zone; Fairbanks, Alaska; Miami, Fla.; State College, N.M.; and Madison, Wis., with panels being removed for testing after 3 months and 1 year of exposure. Panels were exposed when stressed in bending and in the unstressed condition. Test panels were also exposed to several laboratory-controlled exposure conditions in addition to the weathering exposure.

The exterior exposure of panels for 1 year at the Panama Canal Zone has caused an appreciable deterioration in the quality of bonds with 2 of the 5 adhesives, and these same 2 adhesives have also shown some deterioration

during 1 year of exposure at Miami, Fla. There was no deterioration in the bonds exposed for 1 year at the other exposure sites.

The adhesives showing deterioration were of the phenolic-neoprene-nylon and epoxy-resin types, with the deterioration of the phenolic-neoprene-nylon being the more drastic. The phenolic-neoprene-nylon bonds were deteriorated in both the stressed and unstressed condition while the epoxy-resin bonds were not seriously affected, except in the stressed condition.

Laboratory tests consisting of continuous exposure at 120°F and 97 percent relative humidity or a cyclic exposure involving the same temperature and humidity provided results that showed good correlation with the results obtained after exterior exposure in the Panama Canal Zone.

ALLOYS, FERROUS

WADC TR 53-254 (Part 2)

August 1954

SUBJECT: SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH STRENGTH LEVELS
INVESTIGATOR: George Sachs
CONTRACT: AF 33(616)-392
CONTRACTOR: Syracuse University
ABSTRACT: In Part II the information available on fatigue characteristics of high-strength steels is assembled. The data obtained from various sources frequently show great differences even where the testing conditions were presumably identical. Still larger variations resulted from slight differences in testing or in steel conditions the nature of which was frequently unknown.

Contrary to common concepts, high-strength steels may exhibit endurance-strength values considerably superior to those of steels heat treated to a strength of 200,000 psi or lower. This also applies to such values in the presence of stress concentrations. It appears, in general, that any specific steel exhibits fatigue-strength characteristics which may be particularly unfavorable at certain tempering temperatures. High-hardenability high-strength steels usually possess endurance limits considerably above those of other steels heat-treated to high strength levels.

WADC TR 53-254 (Part 3)

July 1954

SUBJECT: SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH STRENGTH LEVELS
INVESTIGATOR: George Sachs
CONTRACT: AF 33(616)-392
CONTRACTOR: Syracuse University
ABSTRACT: Part III of the report presents descriptions of actual failures encountered in high-strength steel aircraft parts and of the experimentation performed for the purpose of analyzing the conditions of failure and determining the sources of it. Practically all of such information relating to aircraft parts has been supplied by aircraft and aircraft parts industries.

The attempt to systematically arrange this information leads to the conclusion that there exists a number of different sources of the failures in high-strength-steel aircraft parts. Furthermore, as a rule a failure resulted from a combination of several factors which deviated from those encountered in regular manufacture rather than from a single factor. From the analysis of the failure cases it may be deduced that a change in one particular factor would have been sufficient and was sufficient in a number of instances to prevent any further failures of the same part.

WADC TR 53-254 (Part 4)

August 1954

SUBJECT: SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH-STRENGTH LEVELS
INVESTIGATOR: George Sachs
CONTRACT: AF 33(616)-392
CONTRACTOR: Syracuse University
ABSTRACT: Part 4 presents a general discussion of the factors which determine the selection of high-strength steels and assembles the data available for their static-strength and design characteristics. In addition, the information available on the effects of the numerous variables encountered in making, shaping and heat treating low-alloy steels and their significance for the strength properties of aircraft parts is discussed.

The regular strength characteristics of the steels, and especially their tensile strength, yield strength, elongation and reduction of area, reveal no indication of an embrittlement of steels heat treated to strength values in excess of 200 ksi, in general, and of those tempered at temperatures between 500 and 750°F, in particular. However, there is some indication that at very low testing temperatures steels heat treated within this tempering range also may exhibit a comparatively low ductility.

The response of static strength properties to other factors which adversely affect their performance, such as section size of slack quenching, is also limited and the practical significance of such effects is not clarified.

WADC TR 53-254 (Part 5)

September 1954

SUBJECT: SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH-STRENGTH LEVELS
INVESTIGATOR: George Sachs, and E. P. Klier
CONTRACT: AF 33(616)-392
CONTRACTOR: Syracuse University
ABSTRACT: Part V summarizes the results of impact tests and notch-tension tests on high-strength steels.

The impact strength of constructional low-alloy steels generally exhibits a minimum at tempering temperatures between 500 and 750°F for short tempering times. Therefore, steels tempered either at about 400°F or over 800°F are preferably used. Also, high-strength steels with optimum impact strength at a given strength level are obtained by holding the carbon

content as low as possible. The effects of the numerous variables encountered in processing and heat treating steels on the impact strength are apparently not universal and not well clarified and frequently are obscured by secondary effects, such as the softening associated with slack quenching.

In contrast, the notch-strength in tension of high-strength steels appears to respond sensitively to a number of variables of different types such as magnitude of stress concentration, eccentricity of loading, section size of quenched and tested specimen, section size of product and method of quenching. The data available at present in these respects, are rather limited but appear to indicate universal trends.

In certain instances a parallelity between impact strength and notch strength has been noted.

WADC TR 53-254 (Part 6)

September 1954

SUBJECT: SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH STRENGTH LEVELS
INVESTIGATOR: George Sachs and E. P. Klier
CONTRACT: AF 33(616)-392
CONTRACTOR: Syracuse University
ABSTRACT: The appendix presents a number of suggestions for future research work on low-alloy aircraft steels heat treated to high strength levels.

WADC TR 53-439

October 1954

SUBJECT: METALLURGICAL TESTING OF BORON STEELS
INVESTIGATOR: C.L. Dotson, J.R. Kattus, and F.R. O'Brien
CONTRACT: AF 33(616)-468
CONTRACTOR: Southern Research Institute
ABSTRACT: Knowledge of the mechanical properties of many medium-carbon, boron-treated steels is insufficient for proper evaluation of the suitability of these steels for aircraft uses.

The hardenability, tensile, impact, notched tensile, and fatigue properties of boron steels 81B40, 98B40, and 81B30 have been determined and are reported. The hardenabilities of carburizing steels 94B17 and 43BV10 have been investigated for different carburized case depths and carbon contents. Impact properties of pseudo-carburized specimens have been determined. Detailed test results are reported for all investigations.

Results of this investigation can be used to determine the suitability of these boron treated steels for aircraft applications by comparison to the properties of alloy steels now used in the aircraft industry.

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ALLOYS, HIGH TEMPERATURE

WADC TR 53-272 (Part 2)

April 1955

SUBJECT: CHROMIUM-BASE ALLOYS
INVESTIGATOR: Haruo Kato and Earl T. Hayes
CONTRACT: PO 33(038)-50-1084 E
CONTRACTOR: U. S. Bureau of Mines
ABSTRACT: The high temperature stress rupture properties of alloys of 60Cr-40Fe to which was added either molybdenum, nickel or cobalt were investigated. Improvements in alloy preparation involving the use of high purity stock and consumable arc melting lowered the oxygen content from approximately 3000 ppm to 300 ppm and improved ductility. These alloys showed very short creep rupture life at 1500°F and 20,000 psi.

WADC TR 53-277 (Part 2)

February 1955

SUBJECT: HIGH-TEMPERATURE PROPERTIES OF FOUR LOW-ALLOY STEELS
FOR JET-ENGINE TURBINE WHEELS
INVESTIGATOR: Adron I. Rush and James W. Freeman
CONTRACT: AF 33(038)-13496
CONTRACTOR: University of Michigan
ABSTRACT: The relationships between type of microstructure and properties at 700°F to 1200°F were surveyed for four low alloyed steels. The steels were SAE 4340, 1.25 Cr - Mo - Si - V ("17-22A"S), 3 Cr - Mo - W - V (H-40), and 13 Cr - Mo - W - V (C-422). Near pure structures were produced by isothermal transformation at a series of temperatures. Martensitic structures were produced by oil quenching. Normalized specimens were also included. Maximum Brinell hardness was kept at 280-320 by tempering the structures which had higher hardness as transformed.

The results indicated that bainitic structures had maximum strength over the temperature range. Tempered martensite in general had intermediate to low strengths. Pearlites were relatively weak at low temperatures, but became similar to the bainites at the higher temperatures. There was considerable variation between high and low temperature bainite and between fine and coarse pearlite. Normalized materials apparently have generally high levels of strength because the usual structures developed are predominantly bainite.

In most cases, rather wide variations in structure were possible with rather uniform properties. There were, however, usually a predominantly strong and an abnormally weak structure within the generalizations. Alloy content controlled the level of strength for a given structure. Thus, while martensitic structures compared unfavorably to the bainites for SAE 4340 and "17-22A"S, the martensitic structure of the C-422 alloy was superior to the lower alloyed steels at the higher temperature and longer time periods. Reasonably good correlations were developed between the structures and properties of turbine wheels of the four alloys on the basis of the results of the survey. The general results from the survey appear to be useful for general guidance in heat treating alloys for high temperature service. However, the survey is very limited and care should be used in extending the data until all the factors have been investigated.

July 1954

SUBJECT: THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS.
INVESTIGATOR: Lawrence A. Shepard, C. Dean Starr, Carl D. Wiseman, and John E. Dorn
CONTRACT: AF 33(038)-11502
CONTRACTOR: University of California
ABSTRACT: Intermittent temperature, constant load creep tests were performed on clad aluminum alloys 75S-T6 and 24S-T3 at 450°F, and the test data are reported herein.

Aluminum alloy 75S-T6 creep specimens were held at 450°F for 1/2 hours out of every two hour temperature cycle. During the remaining half hour of the two hour temperature cycle the specimens were cooled to room temperature, held at room temperature, and then reheated to 450°F prior to the start of the next 2 hour cycle. The 24S-T3 aluminum alloy specimens were held at 450°F for 1 hour out of every two. During the remaining hour of the two hour temperature cycle the specimens were cooled to room temperature, held at room temperature and then reheated to 450°F prior to the start of the next two hour cycle. All specimens were maintained under constant load throughout the test.

Comparison of cyclic temperature creep data obtained in this investigation with isothermal data under the same conditions of stress reveal that approximately equivalent plastic strains are achieved when the comparison is made on the basis of net time at test temperature.

A survey of existing literature on metallic creep under cyclic temperature conditions is also included.

July 1954

SUBJECT: THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS
INVESTIGATOR: Lawrence A. Shepard, C. Dean Starr, Carl D. Wiseman, and John E. Dorn
CONTRACT: AF 33(038)-11502
CONTRACTOR: University of California
ABSTRACT: Tests on the combined effect of intermittent loading and intermittent heating on the creep strength of aluminum alloy 75S-T6 at 450°F were performed.

In all constant and intermittent tests the specimens were initially heated to test temperature in a three hour period. Two types of combined intermittent loading and heating cycles were used after the initial heating and loading which incorporated 1-1/2 hour periods of stressing and heating within a two hour interval. In one type, the "in phase" condition, the time of load application and the acquisition of test temperature were simultaneous.

In the other type, the "out of phase" condition, cooling preceded unloading by 45 minutes.

Within the limits of scatter, simultaneous cycling of the load and temperature in phase produced little effect other than to extend the creep test by the time periods at room temperature and no load. In the case of out of phase cycling of the load and temperature, an acceleration of the creep rate was obtained as compared with isothermal, constant load creep tests assuming that little or no creep should occur at no load, or when the specimen is below test temperature.

WADC TR 53-451 (Part 2)

October 1954

SUBJECT: THE DEVELOPMENT OF A FORGEABLE HIGH-STRENGTH, HIGH-TEMPERATURE, CHROMIUM-RICH, CHROMIUM-IRON ALLOY

INVESTIGATORS: D.P. Moon, H. A. Blank, and A. M. Hall

CONTRACT: AF 33(616)222

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: The second year's work on the development of a forgeable high-strength, high-temperature, chromium-rich, chromium-iron alloy is described. Experimental alloys were produced by induction melting charges of commercially available melting stock, casting into molds, and fabricating by various hot-working methods. The intended composition of the alloys produced during this period was 70 parts chromium, 30 parts iron, 6 to 9 parts molybdenum, 2 to 3 parts titanium, and up to 1/2 part aluminum. This composition had been found, in the previous year's work, to possess excellent stress-rupture properties at 1500F.

The forging qualities of 4- and 12-pound ingots were found to improve as their soundness was improved by the use of preheated alloy charges and slag materials. A portion of this material was successfully hot rolled to thin strip. Little success was achieved in attempts to forge larger ingots. No room-temperature ductility, as indicated by bend tests, was found in rolled strip before or after heat treating. However, the heat treatments affected the hardness and the microstructure. Specimens machined from forged bars of the alloy exhibited remarkable thermal-shock properties when tested at 1800 to 2000 F.

WADC TR 54-120

September 1954

SUBJECT: AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEEL

INVESTIGATOR: Clarence A. Siebert, Maurice J. Sinnott, and Robert E. Keith

CONTRACT: AF 33(616)-353

CONTRACTOR: University of Michigan

ABSTRACT: Specimens from one heat of type 309 - Nb and eight heats of type 310 stainless steel were oxidized in dry, moving air for times up to 100 hr in the temperature range 1600°-2000°F. Intergranular oxidation severity measurements were made microscopically. X-ray powder patterns were made of representative scales. Visual and magnetic examinations were made of both the specimens and the oxide scales. No appreciable difference in the severity of intergranular oxidation

WADC TR 53-373 Sup 2

was observed that could be attributed to differences in alloy content among the heats. In general, intergranular penetration increased with time and temperature. X-ray analysis showed Cr_2O_3 , $\text{Cr}_2\text{O}_3\text{-Fe}_2\text{O}_3$ solid solutions, Fe_2O_3 , and a high-parameter spinel phase in the scales. All of the scales examined were protective in nature, and no improvement in penetration characteristics is foreseen by making minor changes in alloy contents.

WADC TR 54-120 (Part 2)

June 1955

SUBJECT: AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEELS AND HIGH-NICKEL ALLOYS
INVESTIGATORS: Clarence A. Siebert, Maurice J. Sinnott, Lynn H. DeSmyter, Robert E. Keith
CONTRACT: AF 33(616)353
CONTRACTOR: University of Michigan
ABSTRACT: Chromel alloys ASM, ARM, and D, and type 310 stainless steels were oxidized for 100-hour periods in the stressed condition. The above alloys and Inconel were oxidized for times up to 500 hours in the unstressed condition. Intergranular oxidation measurements were obtained microscopically. The influence of stress was to cause an increase in the intergranular penetration when a minimum stress was reached. In general, the intergranular penetration increased with increasing time and temperature. Increasing the water-vapor content of the air increased the intergranular penetration slightly. The effect of a preferred orientation decreased the intergranular penetration slightly. The weight gained during oxidation was determined. It was found that in the alloys tested that a plot of the square of the specific weight gain versus temperature resulted in a straight-line relationship. Visual and magnetic examinations were made on the oxidized specimens and their oxides. No correlation between these observations and oxidation properties could be determined. X-ray diffraction patterns were made on representative oxides. This analysis showed the scales encountered to be of a protective nature by Randall and Robbs criteria of the presence of Cr_2O_3 or high-parameter spinel. Electron diffraction examination of the subsurface structure was performed on type 310 stainless steels oxidized in the unstressed condition. It was found that the oxidation products in the subsurface region were substantially the same as the surface oxides as determined by x-ray diffraction techniques.

WADC TR 54-206

September 1954

SUBJECT: THERMAL-SHOCK INVESTIGATION
INVESTIGATOR: T.A. Hunter, L.L. Thomas, A.R. Bobrowsky
CONTRACT: AF 33(038)-21254
CONTRACTOR: University of Michigan
ABSTRACT: A program of investigation has been undertaken to evaluate the resistance of various materials to thermal shocking. A preliminary analysis of thermal-shock damage has been carried out on a theoretical basis. The results of this theoretical work indicate that the scope of the problem is so wide that purely analytical methods must be supplemented by experimental data.

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An experimental program has therefore been set up to test fourteen materials for their relative resistance to severe repeated thermal shock from the temperature range of 1600 to 2000°F. A suitable apparatus has been constructed and a standard specimen shape has been devised which give reasonable reproducibility of results. Excursions into the subjects of previous specimen history, mechanical fatigue, and thermal wiggling have also been made.

WADC TR 54-276

October 1954

SUBJECT: RESEARCH AND DEVELOPMENT OF WROUGHT AND CAST HIGH TEMPERATURE ALLOYS
INVESTIGATOR: R.R. MacFarlane, R.S. DeFries, E.E. Reynolds, W.W. Dyrkacz
CONTRACT: AF 18(600)-149
CONTRACTOR: Allegheny Ludlum Steel Corporation
ABSTRACT: Study of wrought and cast Co-base and Fe-base alloys was conducted with the object of development of better high-temperature alloys having a minimum strategic alloy content. An alloy containing 10Ni, 10 Cr, 10 W, 5 Mo, and 1 Cb-Ta was outstanding in rupture properties for the wrought Co-base alloys at 1500° to 1700°F. An 18 Mn, 12 Cr, 3 Mo, .8 V alloy had a good combination of properties for application at 1200°F for the wrought Fe-base alloys. Thermal shock properties were best for the cast alloys containing the highest Co. No correlation was apparent between thermal shock characteristics and other commonly measured properties.

WADC TR 54-391

October 1954

SUBJECT: INVESTIGATION ON NOTCH SENSITIVITY OF HEAT-RESISTANT ALLOYS AT ELEVATED TEMPERATURE (RUPTURE STRENGTH OF NOTCHED BARS AT HIGH TEMPERATURES)
INVESTIGATOR: R. L. Carlson, R. J. MacDonald, W.F. Simmons
CONTRACT: AF 18(600)-61
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: Stress-rupture tests were conducted on notched and unnotched or plain bars of S-816, Inconel "X" Type 550, and Waspaloy alloys at test temperatures ranging from 1200°F to 1600°F. The notched specimens had 50 per cent, 60-degree, V-notches with the root radii ranging from 0.005 inch to 0.100 inch. In some tests, as many as three notches of different root radii were used.

The test results indicated that S-816 alloy was notch strengthened by all of the notches used, in the temperature range from 1350°F to 1600°F. Inconel "X" Type 550 was always notch strengthened by all of the notches only at the test temperature of 1600°F. Waspaloy was always notch strengthened by all notches only at the temperature of 1500°F. Both Inconel "X" Type 550 and Waspaloy could be notch strengthened for some test conditions (notch sharpness and time) at temperatures below 1600°F and 1500°F, respectively.

Factors considered to have an influence on stress-rupture behavior have been studied and the results are included. The factors investigated are

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notch geometry, notched and unnotched ductility, the modes of deformation and fracture, metallurgical changes, and surface condition. The influence of some of these factors can vary considerably from alloy to alloy. It does not appear possible, therefore, to evaluate the notch and unnotched stress-rupture behavior of a given alloy completely by any simple method. Rather, an evaluation should be based upon the combined consideration of those factors that are influential in each individual case.

WADC TR 54-451 (Part 1)

November 1954

SUBJECT: EVALUATION OF SURFACE TREATMENTS FOR LOW-ALLOY STEELS
(PART 1. TEST METHOD FOR HEAT-RESISTANT CORROSION
PROTECTIVE COATINGS ON STEEL)

INVESTIGATOR: Sam Tour

CONTRACT: AF 33(616)-406

CONTRACTOR: Sam Tour & Co., Inc.

ABSTRACT: The test procedure involves two types of artificially created environments. The first step is a cyclic or repeated exposure for one week at the desired temperature in an atmosphere of products of combustion. The second step is a cyclic exposure for one week in an alternate condensation corrosion test unit at room temperature or slightly above.

The atmosphere containing products of combustion is obtained by the use of a kerosene torch, kerosene plus suitable additives and an excess of air. The atmosphere produced in this manner is conducted into a full muffle in a temperature controlled furnace. The panels are hung on racks in the furnace muffle where they are heated to the desired temperature for 6 hours each day for five continuous days. During the remaining 18 hours of each day they remain in the closed muffle with no heat applied to the furnace and no artificial atmosphere being introduced into the muffle.

The alternate condensation test equipment has a large turntable revolving about 15 times per hour and on which the specimen panels are mounted. The turntable carries the panels successively through (a) a tunnel where they are cooled with dry air, (b) a tunnel where they encounter warm moist air so as to collect a layer of condensate and (c) an open space where the condensate may evaporate into the room atmosphere.

Sixteen different coatings have been tested. Some of the coatings were tested at temperatures of 600°, 800° and 1000°, others, at 1000°, 1200° and 1400°F in the atmosphere of combustion products of kerosene with additives. Eight of the sixteen coatings mentioned above have been tested at 1000°F in the atmosphere of the combustion products of kerosene without additives.

When the additives were not used, different corrosion effects were observed. These panels withstood the testing conditions in the furnace and in the alternate condensation test better than when the additives were used.

It should be pointed out that this test method is specialized and rather severe; the success or failure of a coating in this test does not imply similar results under other conditions of service or tests.

WADC TR 54-451 (Part 2)

November 1954

SUBJECT: EVALUATION OF SURFACE TREATMENTS FOR LOW ALLOY STEELS.
(PART 2 PAINT CHROMIZING, PAINT SILICONIZING, AND COATING
OF TITANIUM-BORON LOW ALLOY STEEL)

INVESTIGATOR: Sam Tour

CONTRACT: AF 33(616)-406

CONTRACTOR: Sam Tour & Company, Inc.

ABSTRACT: Diffusion coatings produced by chromizing or siliconizing offer considerable promise as heat resistant corrosion protective coatings for use on low to medium carbon, plain carbon or low alloy steels for service at temperatures up to 1200°F. Either of these types of coatings can be applied by the paint process.

The paint used consists of a suspension or slurry containing (a) the desired metal powder (chromium or silicon), (b) a fluoride, (c) glass frits of low and high melting points, (d) a liquid vehicle containing suspension agents and binders, and (e) a volatile thinner.

Substantially no pre-preparation of the steel surface is necessary. The paint is applied by brush, dip or spray. Several coats with intermediate air drying are required. After preheat, the work is heated or fired in an open furnace. Temperatures of 1900 to 1950°F for 1 to 3 hours are required for chromizing. Temperatures of 1750 to 1850°F for 10 to 30 minutes are required for siliconizing. In either case, as furnace heating proceeds, the binders in the paint burn away and the glass frits melt to form a molten protective, but reactive, blanket enveloping the work and protecting it from oxidation as the chromizing or siliconizing reaction takes place. Upon removal from the furnace and cooling, the glass freezes, shrinks and cracks away, leaving the treated surfaces relatively clean.

The low alloy titanium-boron steels can be paint chromized or paint siliconized. The coatings provide protection against scaling during solution heat treatment at temperatures as high as 2100°F.

The chromized cases on low carbon steel are continuous, uniform, ductile layers that withstand bending and forming operations and can be welded. The siliconized cases are hard, brittle and zonal in nature, with a thin interfacial zone that is continuous.

Further development work on both the paint chromizing and the paint siliconizing processes is outlined and recommended.

SUBJECT: DEVELOPMENT OF AUSTENITIC IRON-BASE SHEET ALLOY
INVESTIGATOR: Alfred G. Allten, Edward Sadowski, Allan Simon, and Peter Lillys
CONTRACT: AF 33(616)-2047
CONTRACTOR: Crucible Steel Company of America
ABSTRACT: Two types of high-phosphorus austenitic precipitation-hardening steels were investigated to determine their suitability as sheet alloys for jet engine components. One of the steels which contained approximately 0.4 C, 8.0 Mn, .2P, 8.0 Ni, 19.5 Cr, 1.2 Mo, .2N was tested in sheet form. This steel largely met the objectives of the investigation, in that it contained not less than 50% iron, and was stronger than N-155 at 1200, 1350, and 1500 F. Also, this steel had oxidation resistance only slightly inferior to that of N-155 at 1700F. However, the steel was not readily weldable, and further investigation of this property would be required to properly evaluate the steel.

WADC TR 54-488 (Part 1)

February 1955

SUBJECT: STRESS-RUPTURE, FATIGUE AND NOTCH SENSITIVITY PROPERTIES OF HIGH TEMPERATURE ALLOYS
INVESTIGATOR: Franz H. Vitovec, Benjamin J. Lazan
CONTRACT: AF 33(038)-20840
CONTRACTOR: University of Minnesota
ABSTRACT: Fatigue and stress-rupture data obtained under various combinations of mean and alternating axial stress and static creep data are presented and discussed for S-816 alloy at 75°, 1350, 1500 and 1650°F. Tests were performed under axial stress on unnotched specimens and specimens having theoretical notch sensitivity factors of 2.4 and 3.4. The data are presented as S-N curves and stress range diagrams to show the effect of specimen notch, temperature, alternating-to-mean stress ratio, and stress magnitude on the fatigue and stress-rupture properties.

The role of both creep and fatigue as factors in rupture is discussed with particular reference to temperature and alternating-to-mean stress ratio.

WADC TR 55-23

April 1955

SUBJECT: DEVELOPMENT OF WROUGHT AND CAST ALLOYS FOR HIGH TEMPERATURE APPLICATIONS
INVESTIGATOR: R.R. MacFarlane, R.S. DeFries, E.E. Reynolds, W.W. Dyrkacz
CONTRACT: AF 33(616)2463
CONTRACTOR: Allegheny Ludlum Steel Corp.
ABSTRACT: Developmental studies were conducted on wrought Fe-base and both wrought and cast Co-base alloys for applications at high temperatures. A heat treatable, Fe-base, austenitic alloy containing Mn and Cr was modified with B to give excellent stress-rupture properties at 1200°F. Oxidation resistance was greatly improved by small Al additions. A wrought Co-base

alloy with good stress-rupture properties at 1600 and 1700°F and improved oxidation resistance was developed. Composition levels giving optimum properties were determined for the cast Co-base alloys. Modifications involving B were investigated in both wrought and cast Co-base alloys.

WADC TR 55-96

April 1955

SUBJECT: DEVELOPMENT OF MOLYBDENUM NOZZLE BLADES
INVESTIGATOR: D. V. Doane
CONTRACT: AF 33(600)-23851
CONTRACTOR: Climax Molybdenum Company of Michigan
ABSTRACT: This report describes the development of methods to produce coated molybdenum gas turbine guide vanes (nozzle blades) conforming as closely as possible to USAF Drawing X52D9613. Under this contract 60 vanes have been fabricated and 6 vanes have been coated, using two different coating procedures. The detailed fabrication procedures, coating experiments, and detailed coating procedures are presented in the report.

ALLOYS, NONFERROUS, ALUMINUM

WADC TR 54-104

October 1954

SUBJECT: THE EFFECT OF TEMPERATURE, FREQUENCY, AND GRAIN SIZE ON
THE FATIGUE PROPERTIES OF PURE ALUMINUM
INVESTIGATOR: N. H. G. Daniels, John E. Dorn
CONTRACT: AF 33(038)-22608
CONTRACTOR: University of California
ABSTRACT: A comparison of the fatigue strength of coarse and fine grain high purity aluminum was made at room temperature and at 250°C. The superiority of the fine grain material as evidenced at room temperature was not maintained at the higher temperature where no difference was found between the fatigue properties of the two grain sizes.

A preliminary investigation of the effects of temperature on the fatigue behavior of pure aluminum was also carried out at frequencies of 25 and 1440 cycles per minute. The results suggest that high temperature fatigue of aluminum might be controlled by an activation process with an activation energy of approximately 34 K cals/gm atom, a value closely similar to that found for self-diffusion, creep, stress-rupture, recovery, and grain boundary relaxation. The change of fatigue properties with temperature and frequency is explained in terms of recovery. Supporting metallographic studies showing the corresponding change in structure with particular reference to polygonization have been made.

Other metallographic studies relative to the mode of failure and crack propagation are also described.

WADC TR 54-490

January 1955

SUBJECT: THE EFFECT OF ULTRASONICS ON MOLTEN METALS
INVESTIGATOR: J. Byron Jones, John G. Thomas, Carmine F. DePrisco

CONTRACT: AF 33(616)-2050
CONTRACTOR: Aeroprojects Inc.
ABSTRACT: The application of ultrasonics at a frequency of about 15 kc to small melts of 195 aluminum alloy resulted in accelerated degassing. Grain refinement was observed only when ultrasonic energy was introduced at or above the liquidus temperature. A microphone to appraise elastic energy levels in liquid melts was devised. Extensive investigations of the problems incident to the transmission of elastic vibratory energy into the molten metals were carried out, and the requirements therefore are reported.

WADC TR 54-595

March 1955

SUBJECT: RESEARCH INVESTIGATION OF THE FORGING CHARACTERISTICS OF ALUMINUM POWDER METALLURGY PRODUCTS
INVESTIGATORS: G.D. Dolch, F.E. McBride, J.P. Long, R.A. Paetz
CONTRACT: AF 33(616)2091
CONTRACTOR: Thompson Products, Inc.
ABSTRACT: Three aluminum powder metallurgy products were forged into gas turbine compressor rotor blades to determine if these materials were amenable to such processing.

Forgeability was evaluated through a series of temperatures under various types of working, including: rolling, flat forging, up-setting, extruding, blocking, coining, and trimming.

The effect of forging temperatures on stress-rupture, tensile and fatigue strengths was evaluated by means of specimens prepared from flat forgings. Also, stress-rupture, tensile, and fatigue strengths, along with impact data were obtained on the as-received bar stock for a comparison base line.

Using strength data as a means of appraisal, one material and its optimum forging temperature were selected. Finished compressor blades were processed in accordance with optimum material-forging conditions, and subjected to limited fatigue and metallographic evaluation.

It was found that these materials could be processed into finished compressor rotor blades by substituting extrusion for rolling, and that processing is possible in the temperature range of 500-1000°F.

The two materials with oxide contents in the vicinity of 14% were found to have room temperature strengths comparable to a heat treated wrought aluminum alloy currently used for compressor rotor blades. However, the elongation and impact properties at all temperatures tested were inferior to the wrought aluminum alloy.

ALLOYS, NONFERROUS, MAGNESIUM

WADC TR 54-357

November 1954

SUBJECT: MAGNESIUM SHEET ALLOYS

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INVESTIGATOR: R.R. Nash, H.K. Adams, Jr., A.E. Bibb, Jr. E.J. Tulloch,
M.C. Huffstutler
CONTRACT: AF 33(616)-439
CONTRACTOR: Rensselaer Polytechnic Institute
ABSTRACT: Tentative optimum procedures to add chromium, antimony,
strontium, barium, calcium and manganese to AZ31 base alloy are described.
Relative recoveries of additions of chromium, strontium and barium were extremely
low. Recoveries of antimony, calcium and manganese approached the intended
additions. These additions were made singly and in combination to determine
their influence on the mechanical properties and resistance to corrosion of
AZ31 alloy.

The most promising combination of mechanical strength and ductility was associated with alloys containing single additions of chromium and additions of chromium and manganese together in the as-fabricated by hot rolling condition and after cold rolling a controlled amount followed by a stress relieving heat treatment. Compared with AZ31 alloy on the basis of the same conditions of mechanical working and heat treatment, this combination of mechanical properties was a small but definite improvement.

The relative resistance to corrosion of AZ31 alloy was lowered by additions of chromium, barium, strontium and antimony and improved by manganese additions above a nominal 0.3%. This behavior was related, tentatively, to the iron content of the experimental alloys.

ALLOYS, NONFERROUS, TITANIUM

AFTR 6516 (Part 2)

July 1954

SUBJECT: THE TITANIUM-MANGANESE, TITANIUM-TUNGSTEN, AND TITANIUM-TANTALUM PHASE DIAGRAMS
INVESTIGATOR: D.J. Maykuth, H.R. Ogden, R.I. Jaffee, J.W. Holladay, J.G. Kura
CONTRACT: AF 33(038)-8544
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: Phase diagrams for the binary systems Ti-Mn, Ti-W, and Ti-Ta were determined using both high-purity and commercial titanium.

Manganese shows appreciable solubility in beta titanium, and the beta phase field terminates in a very sluggish eutectoid reaction. Two compounds were found over the alloy range of the Ti-Mn system investigated.

The Ti-W system is characterized, at high temperatures, by a broad beta-plus-tungsten field which originates from a peritectic reaction. At lower temperatures, an alpha-plus-tungsten phase field extends across the diagram as a result of eutectoidal decomposition of the beta phase.

Beta titanium and tantalum are completely isomorphous. Tantalum shows appreciable solubility in alpha titanium. An extensive alpha-plus-beta field exists at low temperatures.

Investigation disclosed that additions at the contaminant level of oxygen or nitrogen to high-purity Ti-Mn alloys have a profound effect on alloy structure.

Data were also obtained on the density, hardness, hot-air oxidation resistance, and mechanical properties of binary titanium-rich manganese, tungsten, and tantalum alloys prepared using iodide titanium.

WADC TR 53-182 (Part 2)

January 1955

SUBJECT: INVESTIGATION OF THE METALLURGICAL CHARACTERISTICS OF THE 36% ALUMINUM TITANIUM-BASE ALLOY
INVESTIGATOR: J. B. McAndrew, D. J. McPherson
CONTRACT: AF 33(616)-196
CONTRACTOR: Armour Research Foundation
ABSTRACT: The second year of work on a project directed toward an assessment of the potentialities of 36% aluminum titanium-base alloys as aircraft structural materials is reported. Experimental data are given concerning the stress-rupture strength of Ti-36% Al at 800°, 900°, 950° and 1000°C; the room temperature and 1000°C tensile strength of Ti-36% Al; the impact strength of the same alloy at room temperature, 700°, and 1000°C; and the oxidation resistance of this material at 1000° and 1200°C in still air environments.

Less extensive data are reported concerning the effects of ternary additions, especially niobium and tantalum, on the properties of the base alloy.

Casting and powder metallurgy techniques applicable to this type of alloy are discussed. Hammer forging was successfully used to prepare material of wrought structure, although this method of forming did not appear useful for the fabrication of finished shapes.

It is concluded that additional research effort directed toward the possibility of modifying the properties of Ti-36% Al to meet the particular demands of various high temperature applications is warranted. Immediate application on other than an experimental basis undoubtedly would be restricted by insufficient development of fabrication processes.

WADC TR 53-230 (Part 2)

June 1955

SUBJECT: STUDY OF EFFECTS OF ALLOYING ELEMENTS ON THE WELDABILITY OF TITANIUM SHEET
INVESTIGATOR: Herbert M. Meyer
CONTRACT: AF 33(616)-206
CONTRACTOR: Armour Research Foundation
ABSTRACT: Three interstitial elements, carbon, nitrogen and oxygen, impair the weldability of titanium alloy sheet. To quantitatively evaluate these influences three typical base alloys containing small interstitial additions were prepared and welded. These alloys were:

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a binary α alloy: 5 Al-Ti;

a ternary alloy containing an α and a β strengthening element: 7 Al-3 Mo-Ti; and

a binary β alloy: 25 V-Ti.

Weldability was to be determined essentially by bend ductility, while other mechanical properties of unwelded and welded sheet were also investigated. Typical heat treatment cycles were to be selected to establish trends for restoring ductility, or strength, or a combination of "optimum" properties.

In the 5 Al-Ti (an α strengthening alloy), tolerance limits proved to be 0.20% for carbon, 0.15% for nitrogen and 0.25% for oxygen. These interstitial contents strengthened the weldment to various degrees.

No general tolerance pattern emerged for the 7 Al-3 Mo-Ti (an alloy containing both an α and a β strengthener). Carbon and nitrogen produced brittle welds under certain specific conditions, but none under others. Oxygen, at the 0.15% level, yields a strong, ductile structure after either a short time, step quench type or a long time, low-temperature anneal.

The 25 V-Ti (not quite a true metastable β alloy) showed little effect from the carbon additions. Nitrogen, which strengthens the weld, did produce erratic ductility results. Oxygen additions displayed no deleterious effect on weldability; as-welded samples were strong and ductile. Oxygen proved to be an excellent weld strengthener and the 0.4% level does not appear to be the limit.

Interesting side developments were the production of a workable and weldable 8 Al-Ti, whereas the 7 Al-Ti was indicated as the forgeable limit previously by many investigators. Also the 15 V-Ti displayed an unorthodox, but promising response to low-temperature isothermal anneals.

Microstructures were methodically used to explain the mechanical properties of the welded zones, and several structural phenomena of major interest were uncovered.

Hardness was not a satisfactory indicator of brittleness in weld zones; it was however, put to good use for controlling the contamination of the α alloy during preparation from the melt.

It may be concluded that interstitial elements in titanium alloy weldments are harmful under some conditions. Where possible, limits of utility have been described. Under some conditions, however, the interstitial elements carbon, nitrogen and oxygen show promise as strengtheners at not too high a cost of ductility, and may be valuable as inexpensive alloying additions already present in the titanium sponge. This fundamental study should help to enlarge and clarify the range of weldable titanium-alloy compositions. At the same time, heat treatments that will restore ductility after embrittlement by the welding cycle are described in terms of the individual phase relationships.

October 1954

SUBJECT: STUDIES OF PHASE RELATIONSHIPS AND TRANSFORMATION PROCESSES OF TITANIUM-ALLOY SYSTEMS

INVESTIGATOR: Donald J. McPherson, William Rostoker

CONTRACT AF 33(038)-8708

CONTRACTOR: Armour Research Foundation

ABSTRACT: Time-temperature-transformation charts have been determined for the following titanium-base alloys, which are of interest for high temperature application: 8% Al-6% Mo, 8% Al-4% Mo, 6% Al-6% Fe, 6% Al-6% Mn and 8% Al-4% Cr.

Alloys in the titanium-rich corner of the Ti-Mo-V system were investigated. The locus of minimum total compositions for the complete retention of beta phase on quenching was determined. Aging studies showed that none of the alloys investigated are permanently β stable. The alloys are evaluated from the standpoint of probable ability to retain ductility under elevated temperature service conditions.

Because of the promising behavior of Ti-Al-V alloys at elevated temperatures, the phase relationships in the titanium-rich corner of this system were determined. The investigation covered the space bounded by Ti-22% Al, Ti-11% V, and 600° to 1200°C. Seven isothermal sections and three selected vertical sections are presented and the phase relationships are discussed.

Phase relationships in the titanium-rich corner of the Ti-Al-Si system were studied for similar reasons. The space bounded by Ti-8% Al, Ti-2% Si and 600° to 1200°C was covered. The diagram is represented by six isothermal and four selected vertical sections.

As a prerequisite for the study of possible age hardening titanium base systems, the solubility limits of silicon, boron, beryllium and carbon in the base compositions Ti-4% V, Ti-3% Mo and Ti-2% Cr were investigated. The carbon-containing alloys were not completed. Information on the other systems is presented in the form of nine partial vertical sections. From this information, selection of the amounts of third component additions and solution treatment temperatures for aging studies will be made.

The formation of two separate and reproducible martensites (both based on a hexagonal lattice) upon quenching binary alloys containing 7 and 9% molybdenum was discovered. No such dual martensitic phases were produced upon similar treatment of Ti-Cr and Ti-Mn alloys.

The method of integrated x-ray diffraction line intensities was proved to be unsuitable for determining the relative amounts of β and α' phases in titanium alloys.

WADC TR 54-101 (Part 2)

April 1955

SUBJECT: STUDIES OF PHASE RELATIONSHIPS AND TRANSFORMATION PROCESSES IN TITANIUM ALLOY SYSTEMS

INVESTIGATOR: D.J. McPherson, W. Rostoker
CONTRACT AF 33(616)-2351
CONTRACTOR: Armour Research Foundation

WADC TR 54-109

March 1955

SUBJECT: SCALING OF TITANIUM AND TITANIUM ALLOYS
INVESTIGATOR: H.W. Maynor, Jr., B.R. Barrett, R.E. Swift
CONTRACT: AF 18(600)-60
CONTRACTOR: University of Kentucky
ABSTRACT: A preliminary study of the scaling characteristics in air of experimentally produced titanium and titanium-base alloys, and commercially - produced titanium and titanium-base alloys was conducted at temperatures of 1200°, 1400°, 1600°, and 1800°F. (650°, 760°, 870°, and 980°C.) in the time range of approximately four to three hundred hours. A total of forty-three titanium-base alloys, one commercial grade of titanium (RS-70), and Type 302 stainless steel were scaled at each of these temperatures; two additional alloys were employed at temperatures of 1200° and 1600°F. Scales formed on a 4.02% Al-Ti alloy were studied in detail and a scaling mechanism was suggested; scales formed on a 4.03% Cr-Ti alloy and a 2.95% W-Ti alloy were studied in less detail. Scaling propensity of titanium-base alloys, relative to titanium and stainless steel, was evaluated on the basis of weight gain with time. Attempts to evaluate scaling propensity on the basis of weight loss with time, through the application of various descaling processes, were unsuccessful; however, results of essentially the same nature were obtained in terms of inches penetration of oxide scale. Isothermal transitions in the parabolic scaling rate were observed for experimentally-produced titanium at 1200°F; transitions were observed, but not studied in detail, for 3.96% Mo-Ti at 1400°F., 1.19% Mo-Ti at 1600°F., and 0.91% Ni-Ti at 1600°F.; transitions were indicated, but not studied in detail, for experimentally-produced titanium at 1800°F.

WADC TR 54-112

September 1954

SUBJECT: INTERMEDIATE TEMPERATURE CREEP AND RUPTURE BEHAVIOR OF
TITANIUM AND TITANIUM-BASE ALLOYS
INVESTIGATOR: Jeremy V. Gluck, James W. Freeman
CONTRACT: AF 33(616)-244
CONTRACTOR: University of Michigan
ABSTRACT: This report summarizes the work performed under Contract No. AF 33(616)-244 during the period from 1 July 1952 to 30 September 1953. The object of the investigation was to determine the relationship between typical structural conditions of representative titanium-base alloys and their mechanical properties in the range from 600° to 1000°F. In order to accomplish this purpose, tensile, short time rupture, and creep properties were determined. The materials studied were: commercially pure titanium, Ti 75A; a commercial alpha-beta alloy, Ti 150A; an experimental stable alpha alloy, 6% Al-94% Ti; and an experimental stable beta alloy, 30% Mo - 70% Ti. In addition, hardness, x-ray, and metallographic studies were made.

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The results indicate that the single phase α or β alloys possess the best combination of properties at 1000°F. Commercially pure titanium, Ti 75A, exhibited lower properties than the alloys at all test temperatures. Wide ranges of strength variation through heat treatment were found for Ti 150A at 600° and 800°F; however, at 1000°F no essential difference between heat treatments was evident, and the absolute level of strength was quite low. The β alloy, 30% Mo, showed little or no response to prior treatment, but had the best combination of creep and rupture properties at 1000°F of the four materials tested. Both Ti 75A and the α alloy, 6% Al, showed improvement in strengths at 600°F from small amounts of cold working. At 1000°F, however, an optimum amount of cold work, less than 10%, existed, above which properties were lowered through metallurgical instability.

WADC TR 54-205

August 1954

SUBJECT: DEVELOPMENT OF IMPROVED TITANIUM-BASE ALLOYS
INVESTIGATOR: Herbert A. Robinson, Paul D. Frost, Walter M. Parris
CONTRACT: AF 33(616)-384
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: The research program during the past year was centered around six alloys which had shown considerable promise in earlier work as potential aircraft structural materials. The tensile properties of the alloys, as affected by variations in hot working procedures and heat treatments, were evaluated.

Outstanding properties were obtained in two alloys:

Ti-3Mn-1Cr-1Fe-1Mo-1V
Ti-5Mn-2.5Cr

Several ingots of the Ti-3Mn-complex alloy were prepared. These ingots have been forged and rolled to bar stock for evaluation by several industrial organizations.

WADC TR 54-205 (Part 2)

June 1955

SUBJECT: DEVELOPMENT OF IMPROVED TITANIUM-BASE ALLOYS
INVESTIGATOR: Herbert A. Robinson, W. Maxwell Parris, Paul D. Frost
CONTRACT: AF 33(616)-384
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: The heat-treated properties of 24 alpha-beta titanium alloys have been evaluated. Manganese, molybdenum, and vanadium were the principal alloying additions. The strength-ductility relationships of these alloys increased with increasing alloy content up to about 7 per cent alloy addition. The Ti-3Mn-complex alloy had room-temperature properties slightly better than those of any of the other alloys tested. However, a Ti-5Mn-2V alloy exhibited excellent elevated-temperature stability at 650F. A binary Ti-8V alloy was very interesting in that it could be quenched from the beta-phase region and aged to give very

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good properties. Most alloys are embrittled as a result of being heated into the beta-phase region.

An evaluation of elevated-temperature properties of the Ti-3Mn-complex alloy indicated that the short-time tensile and 100-hour stress-rupture strengths were improved by heat treating initially to a tensile strength of 180,000 psi, instead of annealing to a level of 135,000 psi. However, there was no difference in the 1000-hour stress-rupture strengths for specimens initially heat treated to 135,000-or 180,000 psi strengths

It was found that pickling to remove the air-contaminated surface of alloy & sheet added large amounts of hydrogen. A process was worked out for descaling and pickling titanium-alloy sheet without appreciable hydrogen pickup. Using this process, the Ti-3Mn-complex alloy sheet has been heat treated to 180,000-psi tensile strength with over 10 per cent elongation in 1 inch.

Increasing the hydrogen content of the Ti-3Mn-complex alloy to 260 ppm decreased its tensile ductility and caused premature stress-rupture failures of notched specimens at room temperature. The hydrogen level that caused embrittlement in this alloy is a function of the heat-treated strength of the alloy. As the strength is increased through heat treatment, the hydrogen content required to cause embrittlement is decreased.

WADC TR 54-280

November 1954

SUBJECT: A METHOD FOR RETAINING BETA PHASE IN THE CORE OF PLATES
AND RODS OF TITANIUM ALLOYS

INVESTIGATOR: P. Herasymenko, J. Winter

CONTRACT: AF 33(616)-2259

CONTRACTOR: . New York University

ABSTRACT: Twelve 1-kg ingots were prepared having the following nominal compositions: Ti-(6,8,10,12%) Cr, Ti-3%Al-(6,8,10%) Cr and Ti-6,9,12%) Mn. The ingots were forged to 1/2-inch and 3/4-inch square bars, which were heat treated using two-step quenching. Microstructures and hardness on the cross-section of bars were investigated.

Evidence is presented that soft beta can be retained in the center of bars and plates by step-quenching in alloys containing 12% Cr in the Ti-Cr series, 10% Cr in the Ti-3%Al-Cr series and 12% Mn in the Ti-Mn series. The outer layers of bars are considerably harder than the core after step-quenching.

WADC TR 54-305 (Part 1)

SUBJECT: HANDBOOK ON TITANIUM

August 1954

INVESTIGATOR: Heinrich K. Adenstedt

CONTRACT: AF 33(616)-2222

CONTRACTOR: Avco Manufacturing Corp.

WADC TR 53-373 Sup 2

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ABSTRACT: Information has been collected on specific properties and procedures of titanium by literature survey and by personal contact with the proper persons and institutions. The data have been evaluated and are condensed in this report. The three major sections of titanium technology covered are: production, physical metallurgy and properties.

WADC TR 54-355

December 1954

SUBJECT: PRECIPITATION HARDENING AND EMBRITTLEMENT OF HIGH-STRENGTH TITANIUM ALLOYS
INVESTIGATOR: H.A. Robinson, M.W. Parris, A.E. Austin, C.M. Schwartz, P.D. Frost
CONTRACT: AF 33(616)-445
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: Representative alloys of the Ti-Cr, Ti-Fe, and Ti-Mo systems were quenched to retain the beta phase and aged at temperatures in the range 500 to 1000 F for periods up to 1000 hours. The changes which occurred in the alloys were followed by means of X-ray diffraction, hardness, and electrical resistivity. From this data it was concluded that the decomposition of the retained beta phase occurred in three major stages:

- (1) Precipitation of a metastable transition phase, omega, which apparently causes age hardening. At the same time the beta phase seems to become locally enriched in alloy. Hardness increased to very high values during this stage.
- (2) The omega phase disappears and the alpha phase makes its appearance. The remaining beta phase becomes highly enriched in alloy. Both hardness and resistivity decrease sharply during this stage.
- (3) In the eutectoid systems (Ti-Cr and Ti-Fe), the highly enriched beta decomposes into alpha plus compound. In the Ti-Fe alloys, this stage was accompanied by a further drop in hardness and resistivity. In the Ti-Cr system there is little change in these properties.

The structure of the omega phase has not yet been determined. However, it was shown to be common to at least the majority of beta-stabilized titanium alloy systems, and was apparently not affected by the interstitial impurity content of the alloys.

WADC TR 54-355(Part 2)

June 1955

SUBJECT: PRECIPITATION HARDENING AND EMBRITTLEMENT OF HIGH-STRENGTH TITANIUM ALLOYS
INVESTIGATOR: W.M. Parris, C.M. Schwartz, P.D. Frost

WADC TR 53-373 Sup 2

CONTRACT: AF 33(616)445
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: The structure of the metastable transition phase, omega, which is involved in the age hardening of titanium alloys, was determined. It is a complex body-centered-cubic cell of the gamma-brass type containing 54 atoms. The phase contains 3.7 atomic per cent of the alloying element. The (100) directions of the omega phase and the parent beta phase are parallel. A tentative mechanism for the age hardening of titanium alloys is proposed.

The formation of the omega phase during aging was found to be inhibited by increasing alloy content. Leaner alloys which transformed to alpha prime upon quenching exhibited some degree of age hardening.

Mechanical-property tests on quenched and aged Ti-4Fe and Ti-8Cr alloys confirmed the severe embrittling effect of the omega phase. Further aging to convert omega to alpha resulted in high strengths with recovery of some ductility.

Attempts to detect the omega phase by metallography or autoradiography were not successful.

WADC TR 54-404

August 1954

SUBJECT: EVALUATION OF TITANIUM AIRCRAFT PARTS SEMI-FINISHED PRODUCTS
INVESTIGATOR: F.J. Gillig, L.W. Smith
CONTRACT: AF 33(616)-471
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.
ABSTRACT: A contact survey of the major commercial producers of aircraft engines and aircraft in the United States was conducted for the purpose of determining the difficulties and problems that have been encountered in the fabrication and processing of titanium and titanium alloys for aircraft applications. Thirty-four companies were contacted, some of which were visited several times.

Upon the completion of the contact survey, the results were assembled and analyzed for trends and outstanding difficulties. Three specific problem areas were selected for further evaluation. In order to obtain additional pertinent information on these problems, questionnaires were sent out to the aircraft companies contacted previously who had reported encountering one or more of the problems. These questionnaires were returned together with test data and reports that substantiated the answers contained therein.

All of the above data have been summarized and are presented in a generalized form in this report.

WADC TR 54-487

March 1955

SUBJECT: THE EFFECT OF GRAIN SIZE ON THE MECHANICAL PROPERTIES OF
TITANIUM AND ITS ALLOYS

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INVESTIGATOR: H.R. Ogden, F.C. Holden, R.I. Jaffee
CONTRACT: AF 33(616)-412
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: A study has been made of the effects of grain size on the mechanical properties of commercial-purity titanium, an alpha-beta-titanium alloy, and a metastable beta-titanium alloy. The mechanical properties studied in this research were tensile, notched-tensile, hardness, bend, impact, and fatigue endurance.

For unalloyed titanium, grain size or shape has only a minor effect on the mechanical properties. Yield strengths are decreased slightly for specimens annealed in the alpha-beta range, because of the partition of impurities to the nonmatrix alpha. Impact resistance is highest for the smallest alpha grain sizes. Bend ductility and tensile ductility are excellent for all grain sizes. The fatigue endurance limit at 10^7 cycles is 79 per cent of ultimate strength for unnotched and 42 per cent for notched specimens.

The grain size of the metastable beta alloy (Ti-7.5Cr-7.5Mo) also has only a minor effect on the mechanical properties so long as the alloy is single phase. The presence of alpha in the structure lowered the bend ductility, tensile ductility, and impact resistance. Notched fatigue properties appear to be relatively unaffected by changes in grain size or microstructural condition. The plates of alpha phase in the beta matrix act as stress raisers similar to notches and lower the unnotched endurance limit.

The alpha-beta alloy (Ti-2.5Cr-2.5Mo) was tested in the stabilized condition so that the only variables would be grain size and grain shape. Only minor changes in properties occurred as a result of changing the alpha-beta grain size. Strengths were unaffected by changing grain shape, although the equiaxed alpha-beta specimens had higher tensile ductilities and impact resistance than the acicular alpha-beta specimens. The fatigue endurance limits generally were unaffected by grain size or shape. The unnotched fatigue strength was 34 per cent of the ultimate tensile strength, and the notched fatigue strength was 20 per cent of the ultimate tensile strength.

Vacuum annealing to remove hydrogen had little effect on the properties because these alloys contained only 30 to 60 ppm hydrogen originally.

WADC TR 54-555

March 1955

SUBJECT: THE EXTRUSION OF TITANIUM
INVESTIGATOR: Alvin M. Sabroff, W. Maxwell Parris, Paul D. Frost
CONTRACT: AF 33(038)-3736
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: Extrusion tests were conducted on unalloyed titanium and the Ti-3Mn-complex alloy to study the effects of extrusion temperature and die design and to evaluate various lubricants and die materials. Optimum mechanical

properties were attained at extrusion temperatures in the alpha-phase region for unalloyed titanium and in the alpha-beta region for the Ti-3Mn-complex alloy.

The surface finish of round bars extruded with flat-face dies was very poor. Improved metal flow and surface finish were obtained with conical dies. The optimum die angle appeared to be about 130 degrees.

Lubricants containing graphite, molybdenum disulfide, and mica produced acceptable surface finishes. The best results were obtained with these materials suspended in a Bentone grease. Titanium carbide, chromium carbide, and cobalt-base alloys showed promise as die materials. The carbides exhibited the least wear and less tendency toward seizing by the titanium. High-quality bars were extruded with these die materials and the Bentone lubricant mixture.

WADC TR 54-609

March 1955

SUBJECT: SPOT-WELDED JOINTS IN TITANIUM ALLOYS AND THEIR BEHAVIOR
IN FATIGUE

INVESTIGATOR: William H. Kearns, Walter S. Hyler, David C. Martin

CONTRACT: AF 33(616)-2005

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: A study of the spot welding of titanium and titanium alloys was made to determine the behavior of six-spot weldments under fatigue loading and to compare the fatigue behavior of similar weldments in titanium, aluminum, and stainless steel. Commercially pure titanium sheet, commercial titanium - 7 per cent manganese alloy sheet, experimental unalloyed titanium sheet, and Type 321 stainless steel sheet, all of 0.040-inch thickness, were used. Static tension-shear and cross-tension tests were made on single-spot welds in the materials and the tension-to-shear ratios were calculated. For equal gages and spot spacing, six-spot joints in the stainless steel sheet were slightly better in fatigue than similar joints in the titanium materials under similar loading conditions. The joints in the titanium materials were significantly better than similar joints in clad 24S-T and 75S-T aluminum alloys. (The data on the aluminum alloys were developed in previous work.) Under static tension-shear loading, the joints in the titanium materials were stronger than similar joints in the stainless steel and the aluminum alloys.

WADC TR 54-616 (Part 1)

January 1955

SUBJECT: HYDROGEN CONTAMINATION IN TITANIUM AND TITANIUM ALLOYS

INVESTIGATOR: Ralph J. Kotfila, Harris M. Burte

ABSTRACT: Hydrogen contamination in alpha-beta titanium alloys can cause low ductility in slow strain rate, room temperature tensile tests, and premature brittle fracture in room temperature rupture tests. The combination of stress concentrations and hydrogen contamination can lead to a drastic decrease in the load carrying capacity of these alloys. Another result of hydrogen contamination

is increased susceptibility to embrittlement as a result of exposure to stress and elevated temperature.

At the present time, a maximum hydrogen content of 125 parts per million is suggested as a tolerance for aircraft quality alpha-beta titanium forging alloys. Data are presented which show that in the future it will be possible to produce alloys which have much higher tolerances for hydrogen.

WADC TR 55-5

May 1955

SUBJECT: TENSILE PROPERTIES AND RHEOTROPIC BEHAVIOR OF TITANIUM ALLOYS AND MOLYBDENUM
INVESTIGATOR: E. J. Ripling
CONTRACT: AF 33(616)-2223
CONTRACTOR: Case Institute of Technology
ABSTRACT: The unnotched and notched tensile properties are described as a function of testing temperature for a series of titanium-nitrogen, and titanium-manganese binary alloys as well as for the commercial alloy, Ti-140A, and the experimental 3 Mn-complex alloy.

It was shown that the nitrogen embrittles alpha titanium by elevating its transition temperature. This brittleness, can be partially eliminated by taking advantage of a rheotropic recovery.

The Ti-140A alloy in the "as-received" condition was high in hydrogen so that a brief investigation of the effect of hydrogen in this alloy was also conducted.

Recrystallization embrittlement in commercial unalloyed molybdenum was found to be a manifestation of rheotropic brittleness.

ALLOYS, NONFERROUS, VANADIUM

WADC TR 52-145 (Part 2)

November 1954

SUBJECT: EXPLORATION OF VANADIUM-BASE ALLOYS
INVESTIGATOR: William Rostoker, Donald J. McPherson, Max Hansen
CONTRACT: AF 33(038)-8517
CONTRACTOR: Armour Research Foundation
ABSTRACT: This report describes the results of work performed during the period May 14, 1952 to May 14, 1953. The forgeability of a large number of binary alloys was investigated. While many proved forgeable, the room temperature ductilities of all but vanadium-titanium and vanadium-zirconium alloys were poor. Ternary alloys based on vanadium-titanium were investigated. Elevated temperature tensile properties have shown much promise.

Studies on improvement of the oxidation characteristics of vanadium alloys have proved fruitful; chromium and aluminum additions were found to be particularly effective. A number of alloys have demonstrated sufficient resistance for 100 hours at 700°C.

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Limited studies were conducted on the systems vanadium-oxygen and vanadium-nitrogen.

WADC TR 52-145 (Part 3)

January 1955

SUBJECT: EXPLORATION OF VANADIUM-BASE ALLOYS
INVESTIGATOR: Albert S. Yamamoto, William Rostoker
CONTRACT: AF 33(038)-8517
CONTRACTOR: Armour Research Foundation
ABSTRACT: This is the third annual report summarizing the results of work performed during the period May 15, 1953 to June 14, 1954. The oxidation study on vanadium alloys has been further pursued and the conclusion has been reached that satisfactory oxidation resistance at elevated temperature of vanadium-base alloys cannot be rendered by alloying alone but possibly by electroplating. Major efforts have been directed toward evaluating the elevated temperature tensile and stress-rupture strengths of the alloys under development. Carbon additions have proven beneficial as far as the forgeability is concerned. In certain cases, however, superior tensile ductilities are observed while no effect in stress-rupture behavior results. The current availability of less expensive vanadium-aluminum alloy produced by the alumino-thermic reduction process has promoted a feasibility study of its use. It is found that the alloys made of this are as competitive in mechanical properties as the equivalent alloys based on the very expensive calcium-reduced vanadium metal.

A range of alloy compositions containing additions of titanium and aluminum have shown stress-rupture behavior which is as good or marginally better than the best titanium-base alloys.

ANALYSIS AND MEASUREMENT

WADC TR 53-201 (Part 5)

August 1954

SUBJECT: THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLTEN MATERIALS
(PART 5 THE THERMAL CONDUCTIVITY OF MOLYBDENUM DISILICIDE
FROM 300° to 800°C)
INVESTIGATOR: E. D. West, D. A. Ditmars, D. C. Ginnings
CONTRACT: AF 33(616)-52-10
CONTRACTOR: National Bureau of Standards
ABSTRACT: A new apparatus is described for determining at high temperatures the thermal conductivities of liquids and solids having relatively high conductivities. The apparatus employs steady state longitudinal heat flow along a rod surrounded by a matched guard tube. Results on molybdenum disilicide are described.

WADC TR 53-373 Sup 2

WADC TR 53-308 (Part 2)

February 1955

SUBJECT: DENSITY AND VISCOSITY OF MOLTEN MATERIALS. PART 2 SUMMARY OF VISCOSITY MEASUREMENTS JANUARY 1952 to AUGUST 1953
INVESTIGATOR: J.W. Sausville
CONTRACT: AF 33(616)52-9
CONTRACTOR: University of Cincinnati

WADC TR 53-308 (Part 3)

May 1955

SUBJECT: DENSITY AND VISCOSITY OF MOLTEN MATERIALS. PART 3 SUMMARY OF VISCOSITY MEASUREMENTS SEPTEMBER 1953 TO AUGUST 1954.
INVESTIGATOR: Curtis C. Beusman
CONTRACT: AF 33(616) 52-9
CONTRACTOR: University of Cincinnati
ABSTRACT: A viscometer was fabricated which uses a torsionally vibrating piezoelectric crystal attached to a metal sensing element. Electronic components for measuring the resonant frequency and the resonant resistance of the damped crystal did not yield adequate sensitivity of reproducibility with the instrument. A servomechanism designed to stabilize the instrument was not successful in remedying the difficulties encountered.

WADC TR 53-378 (Part 2)

December 1954

SUBJECT: THE DEVELOPMENT OF ELECTRICAL CONDUCTING TRANSPARENT COATINGS FOR ACRYLIC PLASTIC SHEET
INVESTIGATOR: George A. Dalin, Ivan Flores
CONTRACT: AF 33(161)-111
CONTRACTOR: Balco Research Laboratories
ABSTRACT: Methods are discussed for the preparation of transparent, conductive films on both acrylic and glass surfaces. A detailed description of the preparation and application of silica undercoatings is presented in addition to sputtering of metallic cadmium under conditions such as to form the oxide. Although the described sputtering techniques apply principally to flat surfaces, the extension of these methods to curved surfaces is discussed. A description of special equipment needed in the sputtering of curved surfaces is also included.

WADC TR 53-455

March 1955

SUBJECT: APPLICATION OF THE POLAROGRAPH TO ANALYSIS OF TITANIUM-BASE ALLOYS
INVESTIGATOR: Philip J. Elving, Charles L. Rulfs, Joseph L. Lagowski, Julian Lakritz, Robert J. Meyer
CONTRACT: AF 33(600)-379
CONTRACTOR: University of Michigan
ABSTRACT: The possible application of the polarographic techniques of direct concentration measurement and amperometric titration to the analysis of titanium-base alloys has been investigated. In view of the limitations of time, budget, and personnel, attention was focused on the development of analytical methods for only three or four elements. A procedure is described for the

simultaneous determination of chromium and estimation of iron in titanium-base materials. The procedure involves a general technique for the dissolution of titanium-rich materials in hydrofluoric acid followed by separation of most of the titanium as insoluble potassium hexafluorotitanate. In the specific procedure described, excess fluoride is then largely removed by an evaporation in the presence of sulfuric acid and the chromium is oxidized to chromate by persulfate. Treatment with ammonia and cyanide serves simultaneously to complex the iron as the soluble ferricyanide and precipitate the residual titanium as hydroxide. The solution is then made strongly alkaline and polarographed. Chromium results are excellent, while those for iron are only fair. A rapid method for the polarographic determination of iron in titanium-base alloys has been developed, using a modified citrate procedure. Hydrofluoric acid is used to dissolve the sample. Aluminum ion is then introduced to complex the fluoride ion and potassium citrate is added as a supporting electrolyte. The polarogram is recorded and the current, after a blank correction has been applied, is compared to a previously determined standardization curve. It is possible to obtain accurate results for iron contents ranging between 0.2 and 5.0%. A complete determination can be completed in 15. to 20 minutes. A rapid and precise amperometric titration method for the determination of vanadium in titanium-base alloys has been developed. Sulfuric acid or hydrofluoric acid followed by sulfuric acid evaporation is used to dissolve the sample. Standard ferrous solution is the titrant and a rotating microplatinum electrode is used as the indicator electrode. Specifically, titanium, iron, manganese, chromium, copper, magnesium, aluminum, and moderate amounts of molybdenum and tungsten do not interfere. In addition, a large number of other elements do not interfere.

WADC TR 54-45

February 1955

SUBJECT: COMPOSITE SPECTROPHOTOMETRIC PROCEDURES FOR THE ANALYSIS
OF LOW-ALLOY STEELS AND OF ALUMINUM ALLOYS.
INVESTIGATOR: S.B. Simmons

WADC TR 54-185 (Part 1)

July 1954

SUBJECT: THERMAL AND RELATED PHYSICAL PROPERTIES OF MOLTEN MATERIALS
INVESTIGATOR: C.T. Ewing, B.E. Boker
CONTRACTS: C.S.O. & A (33-616)52-829
C.S.O. & A (33-616)53-178
CONTRACTOR: Naval Research Lab
ABSTRACT: Thermal conductivity and heat capacity figures for hot-pressed molybdenum disilicide are reported to 840°C. The property measurements were made at the Naval Research Laboratory where work was initiated in July, 1953. The apparatus and method employed for each property study are described in detail. The conductivity coefficients were measured in a longitudinal type system with guard-ring compensation; the heat capacity results were derived from enthalpy measurements made by a drop-method with a copper block calorimeter. A detailed analytical description for each molybdenum disilicide test sample is included,

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and the change in property value from ideal due to the impurity content or physical form of the test sample is predicted, where possible.

WADC TR 54-185 (Part 2)

January 1955

SUBJECT: THERMAL AND RELATED PHYSICAL PROPERTIES OF MOLTEN MATERIALS,
PART 2, HIGH TEMPERATURE REACTIONS OF SODIUM HYDROXIDE
INVESTIGATOR: D. D. Williams, R. R. Miller
CONTRACT: AF 33(616)-54-102
CONTRACTOR: Naval Research Laboratory

WADC TR 54-306 (Part 1)

February 1955

SUBJECT: INVESTIGATION OF THERMAL PROPERTIES OF PLASTIC LAMINATES
INVESTIGATOR: F. R. O'Brien, S. Oglesby, Jr.
CONTRACT: AF 33(616)-2045
CONTRACTOR: Southern Research Institute

WADC TR 54-393

December 1954

SUBJECT: PRELIMINARY STUDY OF GLASS AND PLASTIC FILMS USING ELECTRON
DIFFRACTION AND ELECTRON MICROSCOPY
INVESTIGATOR: L. Harold Bullis
ABSTRACT: Thin films of acrylic plastic prepared from solutions of plastic
in amyl acetate are shown by electron diffraction to be highly crystalline, and the
crystallinity is shown to change with the aging of the films. The crystallinity of
the films and the observed effects of aging are explained on the basis of the
"micelle" theory commonly accepted as the explanation of somewhat similar effects
in rubber. Electron micrographs of the films tend to substantiate this view.

Thin films of silica are shown by electron diffraction to be
devoid of crystalline structure and to exhibit no observable changes upon aging.

WADC TR 54-601

March 1955

SUBJECT: A MODIFIED ADIABATIC CALORIMETER
INVESTIGATOR: Myron W. Belaga, David Coddington, Hyman Marcus
ABSTRACT: A modified adiabatic type calorimeter, capable of measuring the
heat capacities of organic liquids (natural and synthetic) in the temperature
range of 100-500°F, was designed, built, and calibrated.

In this calorimeter a fixed rate of power is supplied to the
sample, and a bath which surrounds the sample container is maintained at the
same temperature in order to prevent heat losses from the sample. The heat
capacity of an unknown material may be calculated from measurements of the time
rate of temperature rise, heat input, and mass of the sample.

The calorimeter has been designed for rapid, simple sample
changing.

The calorimeter was calibrated with materials of known heat
capacity (water, castor oil, and linseed oil), and the following relation between
the calorimeter constant and temperature was obtained:

$$K(t) = 0.256 - 3.706 \times 10^{-5}t + 6.839 \times 10^{-7}t^2$$

BIOCHEMISTRY

WADC TR 53-474

August 1954

SUBJECT: DEVELOPMENT OF AN IMPROVED FUNGICIDAL VINYL COATING FOR COTTON FABRIC
INVESTIGATOR: Richard R. Heitkamp, William J. Dewar, Dudley D. Eichorn
CONTRACT: AF 33(616)-81
CONTRACTOR: Flexfirm Products
ABSTRACT: Secondary plasticizers and extenders were evaluated for their ability to impart fungistatic properties when incorporated into vinyl coatings and applied to cotton fabric. The evaluations were carried out by determining the tensile strength of coated fabric samples before and after two weeks soil burial as described in Federal Specification CCC-T-191a Supplement Section IV, part 5, pages 54 and 55.

The most satisfactory candidate plasticizers and extenders were then formulated into coatings that would meet the requirements of Military Specification MIL-F-4143.

Of all the secondary plasticizers and extenders, only one, dehydroabietylammmonium pentachlorophenoxide, satisfied all requirements in providing the protection from microbial attack and at the same time giving the vinyl formulator the utmost leeway in producing an unlimited color range in waterproof coatings.

Some secondary plasticizers were found to have limited effectiveness where heavy coatings can be tolerated.

WADC TR 54-148

August 1954

SUBJECT: CHEMICAL INVESTIGATIONS OF FLUORINE COMPOUNDS AS FUNGICIDES
INVESTIGATOR: G.C. Finger, F. H. Reed
CONTRACT: AF 33(038)-26990
CONTRACTOR: Illinois State Geological Survey
ABSTRACT: Fifteen aromatic fluorine compounds were prepared for fungicidal screening tests since 1 September 1952. The syntheses and properties of these compounds and intermediates are described in detail except in cases of known or borrowed derivatives. Ten of the test samples are new to the scientific literature. Classes represented are fluorinated benzoic acids, phenols, anisoles, toluenes, biphenols, biphenyl sulfide, and benzyl derivatives.

A search was made for colorless and thermally stable fungicides in the biphenyl and biphenyl sulfide classes. Three solid fluorinated hydroxybiphenyls, also called biphenols, were synthesized with 2,2'-dihydroxy-5,5-difluorobiphenyl showing the most promise. When a sulfur linkage was introduced into the latter to give a biphenyl sulfide, fungicidal potency was increased so that 24-95 parts per million prevented growth of four test fungi.

Cotton thread impregnated with the sulfur compound showed no discoloration or loss in tensile strength, and when treated with 1000 ppm

solution was completely protected. Likewise, 2-fluoro-6-nitrophenol, a potent fungicide, and the difluorobiphenols did not appear to change the tensile strength of the thread.

WADC TR 55-72

April 1955

SUBJECT: A COMPILATION OF DATA FROM EVALUATIONS OF THE FUNGUS RESISTANCE PROPERTIES OF AIR FORCE MATERIALS

INVESTIGATOR: E. L. Hamilton

ABSTRACT: The main object of this work is to provide the designer with a guide for selection of fungus resistant materials in the design and maintenance of Air Force materiel which will require some degree of protection against microbiological degradation.

The materials discussed fall into three general classes: (1) those employing a fungicidal treatment, (2) those without treatment, but which show a natural resistance to fungi because of their chemical composition which does not readily provide fungi with a source of nutrient, and (3) the chemicals or formulations that are toxic to micro-organisms.

Fungicidal treatments which have proven unsatisfactory in the particular formulation tested are also listed. However, those found unsatisfactory may well prove satisfactory when used or tested under other conditions.

Many materials, if selected properly on the basis of future use in combination with other materials in the finished item, may provide a satisfactory fungus resistant material without the necessity of a chemical add-on treatment.

CERAMICS

WADC TR 52-291 (Part 2)

August 1954

SUBJECT: AN INVESTIGATION OF VARIOUS PROPERTIES OF NiAl

INVESTIGATOR: W. H. Herz

CONTRACT: AF 33(038)-10716

CONTRACTOR: American Electro Metal Corp.

ABSTRACT: Work with (NiAl + 5% Ni) has been continued. Stress-to-rupture specimens 9-1/2 in. long have been prepared, and various methods of preparation are discussed.

The addition of "stiffeners" to increase strength at high temperatures has been tried. These additions usually act to decrease the oxidation resistance considerably.

A new aluminide, TiAl, is investigated.

WADC TR 52-291 (Part 3)

April 1955

SUBJECT: AN INVESTIGATION OF VARIOUS PROPERTIES OF NICKEL ALUMINIDE.
PART 3. INVESTIGATION OF THE INTERMETALLIC COMPOUNDS OF ALUMINUM

WADC TR 53-373 Sup 2

INVESTIGATOR: W.H. Herz
CONTRACT: AF 33(038)10716
CONTRACTOR: American Electro Metal Corp.
ABSTRACT: Modification of NiAl by the addition of zirconium or titanium has produced products with improved high temperature properties without decreasing the oxidation resistance.

NiAl + 4% Zr is so far the best, having an excellent oxidation resistance - a weight gain of 2.9 mg/cm^2 after 260 hours in air at 1000°C - a transverse rupture strength of 130,000 psi at room temperature and 160,000 psi at 1100°C , and a stress rupture life of 100 hours at 1000°C and 12,000 psi.

Impact strength of this material varies considerably, needing much development work to achieve consistency. The best results in the NACA drop impact tests are over 15 inch-pounds.

These materials can be handled by hot pressing, cold pressing and sintering, slip casting, hydrostatic pressing, with machining in the pre-sintered state making complicated shapes possible.

A method of homogenizing and purifying gamma TiAl was developed, and the first physical tests were made with this improved material.

WADC TR 53-9 (Part 2)

September 1954

SUBJECT: REFRACTORY MATERIALS FOR USE IN HIGH-TEMPERATURE AREAS OF AIRCRAFT
INVESTIGATOR: Norman R. Thielke
CONTRACT: AF 33(038) 16375 (17284)
CONTRACTOR: Pennsylvania State University
ABSTRACT: During this research period the emphasis on oxide refractory development continued from the previous period; the study of selected carbide systems and examination of thermal shock testing procedures was initiated.

The work with oxide-base materials included investigations of (1) the effects of various metal oxide additions on the crystallographic inversions in SiO_2 , AlPO_4 , and ZrO_2 ; (2) the temperature dependence of the modulus of elasticity determined by the sonic method; (3) the improvement of the mechanical strength and thermal stability of aluminum titanate bodies; and (4) the thermal conductivity, thermal shock resistance, and thermal expansion behavior of aluminum titanate.

Among RO_2 -type oxide additions to SiO_2 , only TiO_2 was effective in suppressing the low-to-high inversion of cristobalite, the temperature being reduced from 225°C to approximately 120°C . Mixtures of AlPO_4 and SiO_4 had lower inversion temperatures than either end member by itself. SiO_2 had little effect on the inversion temperature of ZrO_2 , but the abrupt shrinkage during inversion was substantially eliminated by 0.10 and 0.20 mol substitutions. Additions of TiO_2 and MnO_2 to ZrO_2 lowered the inversion temperature from 1050°C to approximately 850°C .

Curves for the temperature variation of dynamic modulus of elasticity determined by a sonic method were presented for the following materials: 75-S aluminum alloy, SAE 1010 steel, fused silica, Armco iron, soft glass, Linde sapphire, magnesia, titanium dioxide, magnesia spinel, steatite, and a vitrified and semi-vitrified whiteware body.

The effects of the following variables on the strength and thermal expansion of aluminum titanate bodies were investigated: (1) excess alumina, (2) addition of manganese compounds, (3) milling media, (4) addition of soluble ammonium compounds, (5) oxidizing and reducing firing conditions, (6) source of Al_2O_3 and TiO_2 , and (7) reheating. Regardless of firing treatment or any other variable, it was found that the lower the thermal expansion of aluminum titanate the lower the transverse strength and vice versa.

It was shown that aluminum titanate may be stabilized by isomorphous substitutions of iron titanate and magnesium dititanate. Test bodies stabilized by incorporation of 25 weight percent of iron or magnesium isomorphs had low thermal expansion, but also low strength and excessive crystal growth.

The thermal conductivity of aluminum titanate rings was measured, the mean value being approximately $1.11 \text{ kcal/m}^2\text{C.hr.}$ Using the ring test apparatus, the rings were not broken at a ΔT of 428°C , which was the maximum temperature difference obtainable in the apparatus. The static thermal stress resistance of aluminum titanate was shown to be superior to that of other typical ceramic bodies.

Measurement of directional thermal expansion of aluminum titanate by high temperature x-ray powder methods gave coefficients of $+118$, $+194$, and -26×10^{-7} for the a, b, and c coordinate directions in the interval 25° to 970°C . The extreme expansion anisotropy was believed to be responsible for the low strength, low net expansivity, and high thermal shock resistance of aluminum titanate.

The work concerning carbides mainly involved construction of a Gartland-type carbon tube resistor furnace for investigation of compositions in the system TaC-NbC-TiC. It was planned to obtain intercarbide bodies by reactions between carbides, between equivalent metal mixtures and carbon, and between equivalent oxide mixtures and carbon.

It was shown that two factors must be used to characterize the thermal shock resistance of materials, the thermal conductivity, k , and the thermal stress resistivity, $\frac{S}{E\alpha}$. A method for the determination of these factors was described.

The Wright Field simulated service test for turbine blade materials was evaluated in terms of blade failures of seven types.

The predominance of transverse ruptures at a considerable distance from the ends of the blades suggested the possibility of a theoretical treatment of the failures. Thermal stresses in turbine airfoil shapes were approximated by use of a substitute body comprising a plate with rounded edges.

WADC TR 53-37

September 1954

SUBJECT: CERAMIC COATING CONFERENCE 27 and 28 MAY 1952
INVESTIGATOR: Robert J. Brinkman
ABSTRACT: Ceramic coatings are presently being used on various aircraft components such as inner liners, tailcones, turbosuperchargers, etc., for the protection of the metal from oxidation at elevated temperatures.

Various types of laboratory testing procedures for the evaluation of these coatings are presented. Tests for adherence, thermal shock resistance, oxidation and corrosion resistance are a necessity for proper evaluation of the coatings.

Progressive versus single-step testing should be considered in each phase of the evaluation and the variables in each test should be recognized. Quality control measures presently being taken in the production of ceramic coated exhaust components are described. Metallographic examination of the ceramic coating-metal interface for effects of carbon absorption, surface decarburization and intergranular corrosion is another method of evaluating the protective value of the coating.

An outline of the Tentative Testing Procedure decided upon at the conference is included in the Appendix.

WADC TR 53-50 (Part 1)

June 1955

SUBJECT: BEHAVIOR OF BRITTLE-STATE MATERIALS
INVESTIGATOR: O.K. Salmassy, W.H. Duckworth, A.D. Schwope
CONTRACT: AF 33(038)8682
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: The factors influencing the fracture of brittle ceramic materials were studied; the effects of size and stress state were given primary consideration. In addition, initial consideration was given to the effects of strain rate and temperature.

The strength of plaster of Paris was found to decrease with an increase of size in the simple stress states of tension, compression, bending, and torsion. Initial analyses indicated that Weibull's statistical theory of strength could be used to predict the observed effects of size and stress state on the strength of plaster.

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The effects of combined stresses on the fracture strength were studied by means of tests conducted on cylinders of plaster subjected to internal pressure and axial loading. Initial analyses of data from these combined-stress tests indicated that fracture data could be analyzed using the elastic theory of thick-walled cylinders.

The effect of superposed bending stresses on tension-test data was analyzed using Weibull's theory. This analysis indicated that superposed bending stresses should increase the observed tensile strength of a brittle material. Tension data on plaster agreed qualitatively with this prediction.

Analysis of the standard compression test indicated that fracture data from this type of test were unreliable and that the standard compression test could not be used in a research program where precise quantitative fracture data were required.

Exploratory studies were made of the effect of varying the strain rate or the stress rate on the fracture of plaster of Paris. These studies indicated a decrease of fracture stress with increased rates of loading, an effect opposite to that reported in the literature for other brittle materials. The relation between the effects of rate of loading and stress duration (static fatigue) was considered.

WADC TR 53-50 (Part 2)

June 1955

SUBJECT: THE BEHAVIOR OF BRITTLE-STATE MATERIALS
INVESTIGATOR: Omar K. Salmassy, Edward G. Bodine, Winston H. Duckworth,
George K. Manning
CONTRACT: AF 33(038)8682
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: The fracture of brittle-state materials, primarily brittle ceramics, was studied. The principles for the selection, evaluation, and design of brittle materials from a statistical or probability viewpoint were set forth.

The distribution curve of fracture stresses was found to give an adequate description of the strength of a brittle body under a given set of conditions.

The mean fracture stress of a brittle body was found to be inadequate for the design of a brittle structure.

The factors influencing the fracture of brittle ceramic materials were studied, including the effects of size, stress state, strain rate and delayed fracture, temperature, and flaws.

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The entire distribution curve of fracture stresses was indicated to be a function of the size and stress state of a brittle body. All the materials investigated, glass, porcelain, nickel-bonded titanium carbide, plaster of Paris, and embrittled steel showed the same qualitative effect of size and stress state.

Weibull's statistical theory of strength predicted the effect of size, and the effects observed in the simple stress states of tension, bending, and torsion. Weibull's theory was not adequate, however, for predicting the effects of combined stresses.

The research indicated that static-fatigue data will require statistical analysis before they are safe for use in the design of certain ceramics under sustained loads. The materials studied exhibited an increase in strength with increasing strain rate.

WADC TR 53-287

March 1955

SUBJECT: STUDY OF THE SYSTEMS TiC-SiC-B₄C AND TiC-VC-ZrC
INVESTIGATOR: O.E. Accountius, R.F. Stoops, H.E. Konrad, H.M. Greenhouse,
C.C. McBride
CONTRACT: AF 33(038)16911
CONTRACTOR: Ohio State University Research Foundation

WADC TR 54-13 (Part 2)

June 1955

SUBJECT: INVESTIGATION OF THE EFFECT OF RAW MATERIAL PRODUCTION VARIABLES ON THE PHYSICAL AND CHEMICAL PROPERTIES OF CARBIDES, NITRIDES AND BORIDES
INVESTIGATOR: Herman Blumenthal
CONTRACT: AF 33(616)89
CONTRACTOR: American Electro Metal Corp.
ABSTRACT: Section I. Study of Titanium Carbide Powders
The chemical analyses of pure grade commercial titanium carbides were very similar as far as the main constituents were concerned. The differences were only in the oxygen, free carbon and impurity content.

Particle size reduction by ball milling was found to be independent of the ball milling medium and a function only of ball milling time, size and kind of the ball mill, and ball-to-load ratio.

Procedures were worked out for the determination of particle size distribution of a fine powder and for a flotation of such a material, which removed free carbon.

Section II. Properties of Unbonded Titanium Carbide Bars

Titanium carbide powders without binder addition were hot pressed to high densities and tested for electrical resistivity and corrosion resistance.

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It was found that decrease in particle size, addition of up to 1% of free carbon and acid leaching influenced densification beneficially.

Leaching of a ball milled powder before hot pressing or higher densities of the hot pressed bars decreased electrical resistivity.

The only corrosion product found when TiC or TiC/WC 90/10 bars were exposed to air at 1000°C was TiO₂.

Section III. Infiltration of TiC Skeltons

Pieces of low densities were infiltrated with liquid cobalt and nickel and the factors governing infiltrability were studied. It was found that an oxide film surrounding the individual carbide particles was beneficial for good infiltration.

Section IV. Investigation of Bonded Titanium Carbide Bars

Nickel bonded dense titanium carbide bars were produced by hot or cold pressing of ball milled powders followed by vacuum sintering. Various factors determining densification and transverse rupture strength, such as the presence of oxygen, free carbon, iron and other carbides as well as ball milling, pressing and sintering procedures were investigated. It was found that the relationship of these factors to the physical properties of final bars changed considerably with the origin of the titanium carbide used as starting material.

Section V. The Microstructure of Titanium Carbide

A metallographic study was carried out in order to link physical properties to microstructure. It could be shown that microstructure, grain shape and grain growth were functions of three interrelated factors: production procedure of the titanium carbide, surface conditioning of the particles and impurities in the powder. An explanation of the "coring effect", long observed in cemented carbides, was given, based on the assumption of an oxide film surrounding the individual particles.

WADC TR 54-33

April 1955

SUBJECT: PRELIMINARY MICROSCOPIC STUDIES OF CERMETS AT HIGH TEMPERATURES
INVESTIGATOR: E. T. Montgomery, T.S. Shevlin, H.M. Greenhouse, H.W. Newkirk
CONTRACT: AF 33(038) 16911
CONTRACTOR: Ohio State University Research Foundation

April 1955

SUBJECT: THE PRESSURE-CARBONIZATION OF CARBON BONDED SILICON CARBIDE-GRAPHITE FOR USE IN UNCOOLED ROCKET NOZZLES
INVESTIGATOR: B. R. Goss, T.L. Charland, J.R. Tinklepaugh
CONTRACT: AF 33(616)2007
CONTRACTOR: New York State College of Ceramics
ABSTRACT: The resistance to flame erosion of Alfred 410, a carbon bonded SiC-Graphite composition, was improved by the substitution of larger grain sized SiC for the "Settling tank fines" previously used. A new method of forming and firing carbon bonded SiC-Graphite was developed in which the material was carbonized under pressure in stainless steel dies. The process resulted in a product with 15% higher density and 40% lower porosity than that previously obtained. This improvement in properties also resulted in greater resistance to flame erosion.

WADC TR 54-457

March 1955

SUBJECT: DEVELOPMENT OF MATERIALS FOR TRANSMITTING INFRARED ENERGY
INVESTIGATOR: F.W. Glaze, Webster Capps, Douglas Blackburn
CONTRACT: AF 33(616)53-16
CONTRACTOR: National Bureau of Standards

COATINGS

WADC TR 53-73

August 1954

SUBJECT: FIRE-RETARDANT COATINGS
INVESTIGATOR: Sam Collis
ABSTRACT: This project was undertaken in an effort to develop an insulative fire retardant coating suitable for use on aircraft. The desired coating should be a paint-like material, and when applied at a rate of not more than 12 pounds per 100 square feet on metal, should be able to provide a temperature differential of at least 1650°F. for a period of 30 minutes when subjected to a 2000°F flame. In addition, the coating should have the usual properties of a good paint film.

This report covers the initial screening of a number of materials to determine their performance under some of the conditions to which the coating might be subjected. A number of commercially available coatings were evaluated. No entirely satisfactory coating was developed or evaluated. Types or classes of materials deemed worthy of further investigation are noted.

Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they were subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

September 1954

SUBJECT: EVALUATION OF DRY-FILM LUBRICANT COATINGS
INVESTIGATOR: William C. Hart, Bernard Rubin
ABSTRACT: The endurance and load-carrying capacity of commercial and experimental dry-friction reducing films have been investigated at low surface rubbing speeds in the Falex Lubricant Tester. This investigation indicates that the most successful types of dry film lubricants have excellent extreme pressure properties and good anti-wear properties for plain bearing applications at low speeds.

Baked resin-bonded films have superior endurance life than air-drying spray and dip coatings. The endurance life and load-carrying ability of dry film lubricants is dependent upon the resin-bonding agent and pretreatment of the metal surface prior to application of the film. Maximum endurance life is obtained if both surfaces are dry film coated.

WADC TR 53-511

November 1954

SUBJECT: DEVELOPMENT OF A HEAT-RESISTANT RAIN-EROSION-RESISTANT COATING
INVESTIGATOR: F. A. Jeffries
CONTRACT: AF 18(600)-110
CONTRACTOR: Goodyear Tire & Rubber Company
ABSTRACT: High speed flight through rain causes erosion damage to exposed parts of aircraft. Neoprene coatings have been used to protect parts against this damage.

Skin friction due to the high speeds of aircraft and the practice of thermal de-icing, have made it necessary to investigate erosion resistant coatings which will withstand temperatures up to 500°F.

All available elastomers and plastics giving promise of withstanding such temperatures have been examined. Of these, only acrylic ester rubbers, Teflon, and silicone rubbers meet the temperature requirement. Teflon is poor in erosion resistance.

The silicone and acrylic ester rubbers, although they do not have as high a degree of erosion resistance as Neoprene in the present stage of development, afford a fair degree of protection even after exposure to 500°F. Inconsistent adhesion has prevented the ultimate in erosion resistance from being realized.

WADC TR 54-209

November 1954

SUBJECT: SURFACE TREATMENTS OF LOW ALLOY STEELS
INVESTIGATOR: Sam Tour
CONTRACT: AF 33(616)-406
CONTRACTOR: Sam Tour & Company, Inc.
ABSTRACT: This report is to be used as a guide to Air Force Contractors in the selection of surface treatments of low alloy steels for corrosion and

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oxidation resistance at a minimum expense in critical materials. Forty metallic and nineteen paint types of heat resistant coatings applicable to plain carbon and low alloy steels are described and evaluated. Relative temperature, corrosion and abrasion resistances are given. Effects on base metal, formability of coated steel, weldability, joining characteristics and costs per square foot of surface coated are shown. A brief description is given of the processes used for applying and the nature of each of the coatings. Available information and sources of supply are given.

The scope of the protective treatments field is too broad to include all coatings; however, as many coatings were covered as possible. Further, because of the specialized applications involving combinations of elevated temperatures and corrosive environments mentioned in this report, many treatments were evaluated in applications for which they were not intended. Therefore it is not to be assumed that the results tabulated herein are equally valid for other applications or conditions of test.

WADC TR 54-325

January 1955

SUBJECT: RESEARCH FOR LOW-APPLICATION-TEMPERATURE, ELECTRICALLY CONDUCTING, TRANSPARENT COATINGS FOR AIRCRAFT WINDSHIELDS AND RELATED COMPONENTS

INVESTIGATOR: E.R. Olson, P. Schall, Jr., E.H. Layer, E.H. Lougher, R.E. Heiks

CONTRACT: AF 33(616)-342

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: A survey of various methods for producing transparent conducting films at temperatures below 250°F and development studies of one method were conducted in a search for both low- and high-conductivity films. These films are needed for dissipation of static electrical charges and fog, frost, and ice from safety-glass aircraft enclosures.

Of approximately 12 methods investigated, the one chosen for more extensive study was the thermal conversion of metal films to transparent oxides. Evaporated indium films were successfully converted to transparent oxide films at temperatures below the bubbling temperature of the plastic laminate. Films with resistances below 500 ohms per square, measured in vacuum, were produced, but exposure of the films to air at atmospheric pressure caused the resistances to increase by at least two orders of magnitude.

WADC TR 54-373

October 1954

SUBJECT: PROTECTIVE COATINGS FOR MAGNESIUM

INVESTIGATOR: T. Kirk Hay, Gerald F. Bechtle, Garmond G. Schurr, Maurice Van Loo

CONTRACT: AF 33(616)-35

CONTRACTOR: Sherwin-Williams Company

ABSTRACT: The purpose of this investigational work, which was to strive to develop improved coatings for the protection of magnesium, has been accomplished. The recommended finishing systems based on this work use a vinyl toluene-ether

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ester vehicle in both the primer and the topcoat. The primers are pigmented at 35% pigment volume concentration with zinc chromate and silicon dioxide. The solvent resistance of these systems has been variable. The two types used in the samples submitted for evaluation show bad softening but fair recovery upon solvent evaporation.

Time consuming difficulties were encountered in establishing a satisfactory test surface. Pretreatment of the magnesium according to MIL-M-3171, Type III, by a preferred commercial processor and the use of replicate test panels was the solution. Wash primers as pretreatments for magnesium or as an addition to dichromate pretreatments show promise but their performance is too variable to warrant inclusion in the recommended system at this time. Improvements in wash primers included the use of strontium chromate and silicon dioxide as the pigmentation and the reduction of acid activator content to 25% of normal with simultaneous control of water content.

Several items were investigated briefly and showed enough promise to warrant further study. These include polysulfide resins, furan resins, a pigment prepared from calcium sulfide and ammonium vanadate, pigmentation changes in wash primers, and a method for determining the permeability of films to chloride ions.

This work was done by the Paint Research Department of The Sherwin Williams Company under contract AF 33(616)-35 from 29 March, 1952 to 1 June, 1954

WADC TR 54-392

September 1954

SUBJECT: MATERIALS REPORT ON THE LOCKHEED ATC-1 TRANSPARENT ELECTRICALLY-CONDUCTING COATING FOR ACRYLIC PLASTIC

INVESTIGATOR: L. Harold Bullis

ABSTRACT: Accelerated weathering tests, air and rain erosion tests, and flight tests were conducted on the Lockheed ATC-1 transparent, electrically conducting coating to determine its suitability for use on canopies of military aircraft for the reduction of precipitation static. The coating successfully retained the desired values of electrical conductivity, transmittance, and haze throughout most of the tests. On this basis it is recommended from the materials standpoint as a coating meeting the physical requirements established by the Communication and Navigation Laboratory, WADC.

WADC TR 54-452

April 1955

SUBJECT: TRANSPARENT ELECTRICALLY CONDUCTING COATINGS FOR THE REDUCTION OF PRECIPITATION STATIC IN AIRCRAFT

INVESTIGATOR: L. Harold Bullis

ABSTRACT: The Wright Air Development Center program for the development of transparent electrically conducting coatings for the reduction of precipitation static in military aircraft is summarized and evaluated. Work

carried out by Air Force contractors and other organizations is reviewed and discussed. Although a coating meeting all of the desired requirements for aircraft application has not been developed, some coatings, such as the Lockheed ATC-1 showed considerable promise. This coating was flight tested and was able to resist the forces of rain erosion for a considerable period of time without undergoing much change. However, additional flight tests have shown that the degree of static elimination which can be expected by the use of conductive coatings on windshields and canopies is small and does not provide a worthwhile operational improvement. In view of this fact, work on this program has been discontinued.

CORROSION

AFTR 6519 (Part 4)

May 1955

SUBJECT: MATERIALS FOR HANDLING FUMING NITRIC ACID AND PROPERTIES OF FUMING NITRIC ACID WITH REFERENCE TO ITS THERMAL STABILITY

INVESTIGATOR: Mars G. Fontana

CONTRACT: AF 33(038)-10381

CONTRACTOR: Ohio State University Research Foundation

ABSTRACT: Polarization studies were made to study the effect of temperature on galvanic couple systems. These results when used to determine the maximum limiting corrosion rates of the aluminum - stainless steel couple system show good correlation with observed values. RC-70 and Ti-150-A titanium show only a slight decrease in fatigue strength when tested in WFNA. Erosion-corrosion studies show that the corrosion rate increases on aluminum and decreases on stainless steel when exposed to flowing solutions; the effects is more pronounced with increasing rate of acid flow. Results of the "guinea pig" testing program, long time tests in closed containers, are summarized in this report.

A series of measurements of the rate of decomposition and the equilibrium decomposition pressure of pure nitric acid were carried out in glass tubes. The relation between the ratio and vapor volume to the total volume of the sample, the temperature, and the decomposition pressure was established over the temperature range from 76° to 125°C and was extended by extrapolation to room temperature. The initial results of a similar study of the nitric acid - water system are reported. The results when compared with those of pure nitric acid, show that for the same V^G/V and temperature the addition of water in small amounts to the pure acid materially reduces the decomposition pressure and rate of decomposition. The apparatus and experimental techniques used are described in detail.

AFTR 6519 (Part 5)

May 1955

SUBJECT: MATERIALS FOR HANDLING FUMING NITRIC ACID

INVESTIGATOR: Mars G. Fontana

CONTRACT: AF 33(038)-10381

CONTRACTOR: Ohio State University Research Foundation

ABSTRACT: Corrosion fatigue studies were made on RC-70 titanium, titanium alloy Ti-150-A, Armco 17-7PH stainless steel, and 2S aluminum in WFNA (1.5% NO₂) and RFNA (10.5% NO₂) at room temperature. The fatigue life of the titanium and the stainless steel are shortened. Armco 17-7PH was not subject to stress corrosion. Both 3S and 61ST6 aluminum appear to be subject to concentration cell corrosion in WFNA. Polarization data on aluminum and stainless steel agree favorably with actual data obtained from natural galvanic couples of these materials. When aluminum is made anodic to platinum in FNA a phenomenon of anodic passivity occurs at certain values of impressed potential. The corrosion rate of stainless steel can be reduced to a very low value by cathodic protection. These experiments were made using platinum as the inert anode and current densities ranging from 1.5 to 10ma/in² depending on test conditions.

WADC TR 53-16 (Part 3)

December 1954

SUBJECT: CORROSION PREVENTIVE ADDITIVES

INVESTIGATOR: E. J. Schwoegler, L.U. Berman

CONTRACT: AF 33(038)-9202

CONTRACTOR: Armour Research Foundation

ABSTRACT: This project was undertaken to study the development of new inhibitors that may replace or supplement petroleum sulfonates. The work was done by the Organic Chemistry Section of the Department of Chemistry and Chemical Engineering at Armour Research Foundation.

The evaluation of various effective inhibitors against corrosion of SAE 1020 steel in combination with sulfonates has shown that fair inhibition by one sulfonate, is improved by the addition of a small quantity of another inhibitor, usually an amine or amine salts. The amount added was less than that required for good inhibition by either compounds alone.

Over 200 compounds have been evaluated in the JAN-H-792 cabinet. Twelve of these passed the 100-hr requirement. Before using this test method, a study was made to determine reasons for lack of correlation between the results of the Armour Research Foundation cabinet and those of the Wright Air Development Center cabinets. Methods of correlating the cabinet conditions were found so that those compounds listed as passing would also pass the WADC JAN-H-792 test used in qualifying certain specification formulations. Six formulations containing barium dialkyl naphthalene sulfonate, phenothiazine and certain amine salts were also made which passed the 100-hr requirement.

The study of the mechanism of corrosion inhibition as related to structure and functional groups of the polar organic compounds was made on the data obtained from the galvanic system tests with a 52100 steel disk and cartridge brass clip. Most of the information was obtained from the tests conducted at 95% RH at 100°F over a 4-week period in the American Instrument Company cabinet. From the study in MIL-L-6085A base oil the sulfonates, certain amines, amine salts of carboxylic acids, certain oxidized petroleum fractions and certain fatty acid esters of pentaerythritol constituted the effective

structures and groups. In the petroleum base oil, AN-O-6a, sulfonates of lower molecular weight, certain aliphatic amines, polyamine salts of carboxylic acids, certain N-alkyl- and aryl-substituted morpholine salts of carboxylic acids, glyoxalidines, and a few esters were effective.

Several additional compounds were synthesized from data obtained from previous test results but these did not show corrosion-inhibiting properties.

WADC TR 55-84

June 1955

SUBJECT: PACKAGE SAFETY TEST FOR VOLATILE CORROSION INHIBITORS
INVESTIGATOR: R.K. Johnston, J.G. Schafer, L.D. McBeth
CONTRACT: AF 33(616)2119
CONTRACTOR: Nox-Rust Chemical Corp.
ABSTRACT: When a package is assembled using volatile corrosion inhibitors to protect ferrous metal components, there is no known method at present to indicate when the protection drops below a safe level. The development of such package safety tests, which can be applied without opening and re-packaging, was the major objective of this contract. All tests were conducted by personnel of the Nox-Rust Chemical Corporation at their laboratories. The development was carried through the laboratory stage, and resulted in two alternate methods, both based on a rust-inhibition test of the package atmosphere. Various limits of applicability of the two methods have been determined in laboratory tests. These methods are recommended for further study in application to actual packaging operations. Differences in the apparent mode of action of currently available volatile corrosion inhibitors have become apparent during the course of the laboratory work; and it is recommended that fundamental studies be made to determine the mechanisms involved.

CRITERIA DESIGN

WADC TR 53-24 (Part 2)

September 1954

SUBJECT: INTERMITTENT STRESSING AND HEATING TESTS OF AIRCRAFT
STRUCTURAL METALS
INVESTIGATOR: G.J. Guarnieri
CONTRACT: AF 33(038)-10958
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.
ABSTRACT: The high-temperature creep-rupture properties of six sheet alloys having application to aircraft design were investigated under conditions of intermittent load and temperature for comparison with their corresponding constant temperature-constant load behavior. Included are type 321 stainless steel at 1200 and 1350°F, N-155 alloy at 1350 and 1500°F, Inconel X at 1350 and 1500°F, RC-130-A titanium at 800°F, 24S-T3 clad aluminum at 300, 450, and 600°F, and FS-1H magnesium at 300 and 450°F.

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Intermittent load-constant temperature tests were conducted with a cycle of one-hour-load-on, one-hour-load-off in most cases although several of the alloys were subjected also to an eight-hour-on, eight-hour-off load cycle. Intermittent temperature-constant load tests were made using a two-hour cycle with the specimen at temperature one hour and cooled one hour. While these relatively simple cyclic conditions do not duplicate the complex load and temperature patterns encountered in aircraft service, the results obtained do provide some qualitative guidance in applying available static creep-rupture data to design of aircraft structural parts.

A wide variety of effects were produced by the intermittent load and temperature conditions. Stability of microstructure was one of the more significant variables controlling the alloy response to the cyclic conditions. Acceleration of creep and rupture was induced by intermittent loading where such processes as overaging, relaxation, recrystallization, and loss of ductility occurred. Retarding of creep and rupture occurred in those alloys where increase in ductility and creep recovery developed because of the intermittent-load cycle. Intermittent heating produced acceleration of creep and rupture in a number of cases, particularly where susceptibility to intergranular oxidation and cracking was aggravated by thermal stresses.

WADC TR 53-71 (Part 2)

February 1955

SUBJECT: EFFECT OF STRAIN RATE ON THE MECHANICAL PROPERTIES OF
TITANIUM BASE MATERIALS

INVESTIGATOR: J.P. Catlin, W.W. Wentz

CONTRACT: AF 33(038)-21912

CONTRACTOR: Rem-Cru Titanium, Inc.

ABSTRACT: Constant strain rate tensile tests at a variety of strain rates and temperatures were conducted on four commercial-purity alloys representing the three basic types of titanium-base materials (alpha, beta, and combined alpha-beta). The room temperature rate sensitivity ratings, in order of increasing rate sensitivity are:

1. Ti-4%Mn-4%Al (combined alpha-beta).
2. Ti-5%Al-2.5%Sn (alpha).
3. Ti-10%Mn-5%Cr-5%Mo (beta).

Tensile results on high purity unalloyed materials containing individual additions of C, O, N, and H indicate that the C, O, and N additions (0.3, 0.2 and 0.1% respectively) cause an increase in rate sensitivity at room temperature. The hydrogen addition (0.01%) had little effect on rate sensitivity.

The above interstitial additions, nitrogen excepted, also minimized the extent of twinning during deformation. Some evidence of increased amounts of twinning with decreasing strain rates was also obtained.

February 1955

SUBJECT: EFFECT OF STRAIN RATE ON THE TENSILE PROPERTIES OF SAE 4340
STEEL

INVESTIGATOR: Richard F. Klinger

ABSTRACT: The effect of tensile loading rate was determined at room temperature on SAE 4340 steel ranging from the annealed condition to a nominal ultimate strength of 220,000 psi. Stress-strain curves were obtained and the mechanical properties determined over a strain rate range of from 0.00002 in/in/sec to 20 in/in/sec. The results indicate that the strength properties at the higher strain rates are greater than at the low strain rates and that this strain rate effect is less as the strength level of the steel is increased. This trend continues to the 220,000 psi level which shows no change or a slight loss in strength at the higher strain rates. The elongation was not affected by the change in strain rate at any strength level tested.

WADC TR 54-122

February 1955

SUBJECT: EFFECT OF STRAIN RATE ON THE STRENGTH PROPERTIES OF SINGLE AND
MULTIPLE RIVETED LAP AND BUTT JOINTS

INVESTIGATOR: Richard F. Klinger

ABSTRACT: Slow and rapid loading tensile shear tests were conducted at room temperature on single and multiple rivet lap joints and also on butt joints with multiple riveted cover plates. The rivets were made of 24S-T31 and Al7S-T3 aluminum alloys in 1/8 inch and 3/16 inch diameters. The sheet material used was 0.064 inch and 0.072 inch clad 75S-T6 aluminum alloy. The joints were fabricated by standard machine riveting and shop practice. The ultimate strengths of the joints were determined with times to the ultimate load of 0.03 seconds and one minute. All failures were by rivet shear. No pronounced effect of rapid loading on the ultimate strength of any of the riveted joints tested was found although a slight trend toward decreasing strength was observed.

WADC TR 54-175 (Part 1)

August 1954

SUBJECT: NOTCH SENSITIVITY OF HEAT-RESISTANT ALLOYS AT ELEVATED
TEMPERATURES

INVESTIGATOR: Howard R. Voorhees, James W. Freeman

CONTRACT: AF 18(600)-62

CONTRACTOR: University of Michigan

ABSTRACT: Tests have been performed seeking to understand the factors affecting notch sensitivity of heat-resistant alloys under sustained loads at elevated temperatures. The investigation was based on the belief that varied response to notches must be related to relaxation characteristics of alloys at service temperature. A material was postulated to be strengthened or weakened by a notch according to the portion of total rupture life consumed while initial stress concentrations around a notch are reduced and redistributed by a creep-relaxation process. A procedure was proposed whereby the history

of representative fibers in a notched specimen would be followed to the point of rupture.

Data from other sources comparing strengths for smooth and notched bars of materials of interest are included. Additional data required for the proposed analysis were obtained under the present program for three alloys with conventional heat treatments:

S-816 at 1350°F
Waspaloy at 1500°F
Inconel X-550 at 1350°F

Test results included stress - rupture time properties, short-time tensile properties, and creep properties when stresses were changed from one level to another during a test. Relaxation characteristics were measured for initial stresses both below and above the proportional limit.

The notch strengthening observed for S-816 and Waspaloy, and the notch weakening for Inconel X-550 at the test temperatures has been satisfactorily explained in terms of comparative relaxation and stress-rupture time characteristics, though further work is indicated before a quantitative correlation is attempted.

Tests were conducted to determine the effect of some metallurgical variables on the notched bar rupture test characteristics. Cold working had the greatest effect on notch sensitivity of the several conditions investigated, but no severe case of notch weakening was observed for either S-816 at 1350° and 1500°F, or for Waspaloy at 1500°F in the limited number of tests.

WADC TR 54-214 (Part 1)

February 1955

SUBJECT: ELEVATED-TEMPERATURE TESTING PROCEDURES. PART 1. CONTINUOUS RECORDING OF TIME-DEFORMATION READINGS DURING CREEP-RUPTURE TESTING AT TEMPERATURES UP TO 1200°F

INVESTIGATOR: William H. Rector, Charles A. Townsley

ABSTRACT: A new method of recording deformation during creep-rupture testing at temperatures up to 1200°F is presented. This method has the advantages of:
(1) obtaining a continuous time-deformation record up to failure, compared to the intermittent readings obtained using the former manual method and
(2) being completely automatic and saving the technician's time required to make manual measurements. This method employs cameras to record the data. A description of the equipment is given.

WADC TR 54-402

September 1954

SUBJECT: CREEP BUCKLING OF COLUMNS
INVESTIGATOR: D. Rosenthal, D. Hasanovitch
CONTRACT: AF 33(616)-379

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CONTRACTOR: University of California

ABSTRACT: An upper and lower bound to the exact solution of creep buckling has been evolved. In both cases time-deflection curves are computed using isochronous curves which are cross plotted from creep tension data. In addition to the usual assumptions of the beam theory the solution is based on the assumption that the variation of stress in the cross section can be approximated by a parabola in terms of the position in the plane of bending. Under these assumptions the method of solution is quite general and applicable to prismatic columns of any type and initial eccentricity. Comparison with experimental data on Stabilized 24S Aluminum Alloy suggests that the upper bound is a closer approximation to the solution than the lower one. There is also an indication that for moderately crooked columns subjected to high average stress the tangent modulus to the isochronous curve at the average stress level would provide a closer lower bound than the one given by the theory. The deflection at a given time could then be determined directly from the isochronous curve at this time. However, the justification for this procedure could not be established theoretically and it must come from the experiment.

An attempt to extend the theory to columns with high slenderness ratio shows that the solution depends critically on the initial eccentricity, a result which is in qualitative agreement with the experiment.

The investigation shows a need for further work, both experimental and theoretical. In particular, there is a need for duplicate tests to determine the scatter of experimental data.

WADC TR 55-18 (Part 1)

May 1955

SUBJECT: DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS-CONCENTRATIONS AND HYDROGEN EMBRITTLEMENT. PART 1. EFFECTS OF HYDROGEN EMBRITTLEMENT ON HIGH STRENGTH STEELS-STATIC PROPERTIES

INVESTIGATOR: E.P. Klier, B.B. Muvdi, George Sachs

CONTRACT: AF 33(616)2362

CONTRACTOR: Syracuse University

ABSTRACT: In Part I data on the hydrogen embrittlement of one heat of 4340 steel are presented and evaluated. The data represent the mechanical behavior of the embrittled steel in the presence of stress-concentrations. Two different methods of embrittling the steel were employed. The first was to cathodically embrittle the specimens in a bath of 10 percent NaOH, and the second was to introduce embrittlement through the commercial plating of the specimens.

Three different tests were performed to evaluate the effect of hydrogen embrittlement on the steel under different test conditions. The first was a tensile test and the second a bend test. In both of these tests several loading rates were used to evaluate the effect of embrittlement under different loading conditions. The third test performed was a stress-rupture test to render the evaluation of embrittlement under sustained-load applications possible.

Several of the tests examined are suitable for the evaluation of hydrogen embrittlement. However, the bend test is the simplest, and most economical of these tests. At the same time it is a sensitive method of evaluating hydrogen embrittlement in ultra-high strength steels.

ELECTRODEPOSITION

WADC TR 54-240

September 1954

SUBJECT: A STUDY OF CADMIUM-TIN AND ZINC-TIN ALLOY ELECTRODEPOSITS
INVESTIGATOR: Bennie Cohen
ABSTRACT: A study has been made of cadmium-tin and zinc-tin alloy electrodeposits in the continuing efforts of the Air Force to find superior corrosion resistant coatings. The alloy systems evaluated were electrodeposited from fluoborate solutions. Cadmium was used as a basis of comparison throughout. The cadmium-tin alloy coating was found to be superior to cadmium coatings in the majority of tests performed. The cadmium-tin alloy coating was found to have excellent resistance to salt spray, jet fuels, high temperature synthetic oils, organic acid vapors, and to have very little embrittling effect on hardened steel.

WADC TR 53-271 (Part 2)

March 1955

SUBJECT: DEVELOPMENT OF A SUBSTITUTE ON IMPROVEMENT OF CHROMIUM ELECTRODEPOSITS
INVESTIGATOR: Jesse E. Stareck, Angelo C. Tulumello, Edgar J. Seyb, Jr.
CONTRACT: AF 33(616)234
CONTRACTOR: United Chromium, Inc.
ABSTRACT: The effect of a chromium deposit on the fatigue limit of chromium plated, heat-treated steel is correlated with the stress possessed by the chromium deposit. High tensile stress results in severe lowering of the fatigue strength of steel and conversely low stress gives much less fatigue strength lowering.

Numerous stress measurements with both the "spiral contractometer" and "rigid flat strip" methods showed that the stress as measured by the two methods is different for the same deposits, the "rigid flat strip" method giving a lower stress value. Stress changes rapidly with chromium thickness and is correlated with the formation of a crack pattern in a deposit. Deposits possessing fine crack patterns showed compressive stress at thicknesses beyond a few thousandths of an inch.

The baking of a chromium deposit, as commonly practiced to relieve hydrogen embrittlement of plated steel severely lowers the fatigue strength for the usual electrodeposited chromium. This fatigue strength lowering is again correlated with an increase in stress which is due to a shrinkage of the deposit during baking.

This fundamental knowledge about the mechanism of fatigue in chromium plated steels led to the development of baths and plating conditions

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which give greatly improved fatigue properties for the unbaked plate and also other deposits giving equally good fatigue properties after baking.

WADC TR 54-485 (Part 1)

February 1955

SUBJECT: ELECTRODEPOSITION OF TITANIUM
INVESTIGATOR: Walter E. Reid, Jr., Joseph M. Bish, Abner Brenner
CONTRACT: AF 33(616)-53-11
CONTRACTOR: National Bureau of Standards
ABSTRACT: Numerous non aqueous solutions were investigated in an attempt to electrodeposit titanium. Some work was also done with zirconium. Ether solutions containing halides, hydrides, borohydrides, and organo-metallic compounds of titanium were the most promising solutions investigated. A mixed type of bath containing both hydrides and borohydrides yielded titanium-aluminum alloys containing about six percent titanium. Similar baths containing zirconium, instead of titanium, were studied. The zirconium baths gave alloy deposits containing up to 45 percent zirconium.

New methods of preparation of titanium and zirconium borohydrides were developed.

WADC TR 54-485 (Part 2)

April 1955

SUBJECT: ELECTRODEPOSITION OF TITANIUM
INVESTIGATOR: Walter E. Reid, Jr., C. Agnes Gaudette, Abner Brenner
CONTRACT: AF 33(616)-53-11
CONTRACTOR: National Bureau of Standards
ABSTRACT: The study of titanium-aluminum alloy baths as described in WADC TR 54-485 Pt. 1 was continued.

The preparation of concentrated titanium-aluminum and zirconium-aluminum alloy baths by other means than use of the difficultly obtainable borohydrides of titanium and zirconium was investigated. This was done by reacting in an ether solution boron trichloride, lithium aluminum hydride, aluminum chloride, and titanium or zirconium tetrachloride. Under the experimental conditions used, this method was not successful.

An investigation was made of the possibility of using alcohols as complexing agents in solutions of titanium halides. No metallic deposits were obtained from this type of bath.

The unavailability of lithium borohydride (used to prepare titanium and zirconium borohydrides) from commercial sources made necessary an examination of methods for preparing this compound and a quantity was prepared.

FATIGUE

WADC TR 53-437 (Part 2)

October 1954

SUBJECT: THE INFLUENCE OF SURFACE TREATMENT ON THE FATIGUE PROPERTIES OF TITANIUM AND TITANIUM ALLOYS

INVESTIGATOR: Lars Thomassen, Maurice J. Sinnott, Albert W. Demmler, Jr.
CONTRACT: AF 33(616)-26
CONTRACTOR: University of Michigan
ABSTRACT: The fatigue properties of a commercial all-alpha alloy, an experimental all-alpha alloy, and an experimental all-beta alloy, as determined by a R.R. Moore rotating beam type of test, have been evaluated as a function of three different surface conditions. Shot peening improves the fatigue life while grinding decreases the fatigue life, as compared to a hand finished surface. The extent of the increase or decrease varies with the fatigue life range.

Notch fatigue properties of alloys Ti-75A, RC-130B, A-110AT, and experimental all-alpha and all-beta alloys have been determined. Notch types and methods of notch preparation have been evaluated. Rolling operations produce notches that do not adversely affect the fatigue lives. Grinding of notches, either carefully controlled diamond grinding or commercial grinding, very markedly decreases the fatigue life.

WADC TR 54-200

July 1954

SUBJECT: PRELIMINARY INVESTIGATION OF THE EMANATION FROM COLD-WORKED METALS
INVESTIGATOR: F. H. Vitovec
CONTRACT: AF 33(038)-20840
CONTRACTOR: University of Minnesota
ABSTRACT: The disturbance of metal surfaces causes an emanation which can be detected by either Geiger-Mueller counters (Kramer effect) or by photographic emulsions (Russell effect). It was found that the photographic action is caused by the formation of hydrogen peroxide. Various explanations of the origin of the Kramer effect have been attempted, but none of them are completely satisfactory.

WADC TR 54-462

March 1955

SUBJECT: AN ANALYSIS OF NOTCHED UNIAXIAL FATIGUE DATA
INVESTIGATOR: William E. Dirkes
ABSTRACT: An analysis of the failure trends of fatigue data is presented in terms of the nominal applied stresses. Fatigue failure stresses are related to the maximum tensile stresses applied to test specimens. The concepts presented are used as a means of expressing notched specimen test results as functions of unnotched specimen characteristics.

Test data for 2014-T6(14S-T6) and 2024-T4(24S-T4) aluminum alloys and SAE 4130 steel were taken from WADC TR 52-307 and NACA TN 2639 for use in this analysis. The test data available are in agreement with the concepts presented; however, additional data are required to prove the analysis.

Data for failure of unnotched fatigue specimens are used as the

basis for the development of a method for predicting fatigue test data for notched specimens by a semi-graphical method. Fatigue strengths for notched specimens can be predicted for various mean strengths from tensile test data, fatigue data for unnotched specimens, and a single SN curve for notched specimens.

The method as presented is limited to ductile materials tested with axially applied loads and is applicable for fatigue strengths determined for large numbers of cycles.

FUR

WADC TR 53-333

July 1954

SUBJECT: A STUDY OF SYNTHETIC FUR RUFFS

INVESTIGATOR: R. M. Ellis, Jr.

ABSTRACT: Laboratory and field tests on synthetic wolverine furs were conducted at Wright Air Development Center, Wright-Patterson Air Force Base, Ohio, and in Alaska, respectively. The laboratory tests were conducted on sixty constructions representing variations in methods of fabrication and fiber composition. The furs for field tests represented eight different variations of the most promising types used in the laboratory evaluation. Natural wolf fur was used for comparison in both evaluations.

In both laboratory and field tests the better synthetic furs were superior to the wolf fur in ice shedding ability. In addition, the synthetic fur is domestically and readily available and should cause no storage problems.

In wind protection and comfort to the wearer the natural fur appeared to be superior.

The tests have provided data which should be useful in designing synthetic fur which will be satisfactory in every respect. The importance of comfort is emphasized.

JOINING

WADC TR 52-322 (Part 3)

August 1954

SUBJECT: CAUSES OF CRACKING IN HIGH-STRENGTH WELD METALS

INVESTIGATOR: A.J. Jacobs, R.P. Sopher, P.J. Rieppel

CONTRACT AF 33(038)12619

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: This report summarizes the third year of experimental work at Battelle on the causes of cracking in high-strength weld metals. During the period from August 12, 1953, to August 12, 1954, hot-tension and weld-metal cracking studies were conducted on SAE 43XX-type steels and other selected steels. Results from these studies showed a correlation, inasmuch as an

increase in carbon, sulfur, and phosphorus tended to lower hot ductility and promote hot-crack susceptibility, and a misch metal addition seemed to have the opposite effects.

WADC TR 54-17

August 1954

SUBJECT: JOINING OF MOLYBDENUM

INVESTIGATOR: W. N. Platte

CONTRACT: AF 18(600)114

CONTRACTOR: Westinghouse Electric Corporation

ABSTRACT: By the use of a closed chamber and a continuous flow of gas through this chamber a controllable welding atmosphere has been provided for experimental work. The atmosphere may be varied from 100% argon, 99.95% pure, to 100% O₂, N₂ or any combination of these or other gases. The chamber provides a fresh gas shield in an atmosphere of the same composition as the shield.

Using the controlled atmosphere chamber, welds in carbon deoxidized arc cast molybdenum are shown to be subject to hot short cracking when the oxygen content is the argon atmosphere around the arc exceeds 0.2%. The ductility of these welds was drastically reduced by the presence of more than 0.05% oxygen in the welding atmosphere.

Oxygen in sintered molybdenum is shown to produce porosity and hot short cracking. However, crack and porosity free welds have been produced by using deoxidizing agents in sintered molybdenum.

WADC TR 54-97

September 1954

SUBJECT: STUDIES AND EXPERIMENTAL INVESTIGATIONS ON THE FLASH WELDING OF ALLOYED STEELS

INVESTIGATOR: Ernest F. Nippes, Warren F. Savage, Gordon Grotke, Salvatore M. Robelotto

CONTRACT: AF 18(600)-71

CONTRACTOR: Rensselaer Polytechnic Institute

ABSTRACT: Weld centerline rates of cooling at 1300, 1000, and 900°F were determined as functions of three welding variables, the rate of platen acceleration, the final clamping distance, and the thickness of the material, for flash welds employing parabolic flashing patterns. Three rates of platen acceleration, 0.120, 0.042, and 0.0166 in./sec.², were investigated, with a range of final clamping distances of 0.3 to 1.2 in., for rectangular bar material of 0.250 and 0.375 in. thickness.

Flash welding variables within these limits resulted in weld centerline cooling rates, at 1300°F, ranging from a minimum of 40°F/sec. to a maximum of 365°F/sec. Increasing platen acceleration, increasing section thickness and decreasing final clamping distance resulted in an increased rate of cooling. In both alloy steels studied, AISI 4130 and 4340, the formation

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of considerable amounts of martensite at the weldline could not be avoided by any reasonable adjustment of flash welding variables studied.

Flash welds were prepared using AISI 1020, 4130, and 4340 steels at four predetermined rates of cooling. Photomicrographs and the results of hardness and tensile tests are presented and discussed to relate the weld properties and the weld-zone micro-structures.

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April 1955

SUBJECT: STUDIES AND EXPERIMENTAL INVESTIGATIONS ON THE FLASH WELDING OF ALLOYED STEELS
INVESTIGATOR: Ernest F. Nippes, Warren F. Savage, Gordon Grotke, Salvatore M. Robelotto
CONTRACT: AF 18(600)-71
CONTRACTOR: Rensselaer Polytechnic Inst.
ABSTRACT: Weld centerline rates of cooling at 1300, 1000 and 900°F in flash welds in AISI 1020 and 4340 steels, prepared under similar conditions, were measured and compared. The cooling rate is not influenced by the composition of the steel if the specimen geometry, platen acceleration, and final clamping distance are maintained constant.

Weld zone cooling rates were determined for welds in AISI 1020 steel subjected to 1.5 sec. of upset current at an average current density of 18,500 amp/in.². The reduction in cooling rate produced by this upset current flow was obtained by comparison with previous data on welds without post-upset current.

The temperature distribution in the vicinity of the weld zone was measured experimentally during the application of 4.75 sec. of temper current at an average density of 21,400 amp/in.².

Three steels of differing elevated-temperature strength were employed for welds prepared at four rates of platen acceleration to determine the influence of the composition of the steel and the influence of the temperature gradient, established during flashing, on the upset force requirements.

WADC TR 55-22

April 1955

SUBJECT: OXIDATION RESISTANT BRAZING ALLOYS
INVESTIGATOR: George H. Sistare, Jr., Allen McDonald
CONTRACT: AF 33(616)-2205
CONTRACTOR: Handy and Harman
ABSTRACT: During the period from 30 June 1953 to 31 December 1954 some 117 experimental brazing alloys were prepared at the Handy and Harman research laboratories, and evaluated as possible candidates for joining heat conducting

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metal fins to inconel tubing for service at temperatures in the order of 1400°F to 1600°F where oxidation resistance of the joint was mandatory.

Alloys of gold-nickel-chromium, and palladium-nickel base alloys with chromium and silicon were developed which can be used to join stainless steel to inconel at brazing temperatures in the range of 1900°F to 1950°F. The alloys can be torch brazed using flux, or used without flux in a protective atmosphere. The resulting joints resist oxidation at 1600°F service temperature.

It is still possible that with further development palladium-nickel base alloys with silicon-phosphorus-boron additions can be produced which will braze in the temperature range from 1850°F to 1900°F.

The report also contains a note on graphite brazing.

LEATHER

WADC TR 54-63

September 1954

SUBJECT: DEVELOPMENT OF LEATHER FOR MECHANIC'S WINTER GLOVE
INVESTIGATOR: Lewellyn G. Picklesimer, William T. Roddy
CONTRACT: AF 33(600)-22172
CONTRACTOR: University of Cincinnati
ABSTRACT: A comparison of the properties desired in glove leather tannages for a comfortable mechanic's winter glove was made. The data collected indicate that irrespective of tannage the leather has a high rate of oil absorption.

The application of various impregnants to glove leather gave an improvement in oil resistance. Some of the impregnants were very oil resistant but did not penetrate into the leather. Using such impregnants as finish films on the leather would give oil resistance but when the finishes were abraded through, the leather would offer very little oil resistance. The data obtained indicate that it is possible to prepare a glove leather for winter mechanic's wear which will have good resistance to oil absorption without materially reducing the desirable properties inherent in glove leather.

WADC TR 54-466

June 1955

SUBJECT: PRODUCTION OF LEATHERS RESISTANT TO POWERFUL OXIDANTS AND REDUCTANTS
INVESTIGATOR: N.D. Cheronis, M.A. Wentz
CONTRACT: AF 33(600)-23872
CONTRACTOR: The Synthetical Laboratories

METALS, GENERAL

AFTR 6731 (Part 3)

May 1955

SUBJECT: SHORT-TIME CREEP PROPERTIES OF STRUCTURAL SHEET MATERIALS FOR AIRCRAFT AND MISSILES

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INVESTIGATOR: John A. Van Echo, W.F. Wirth, Ward F. Simmons

CONTRACT: AF 33(038)-8743

CONTRACTOR: Battelle Memorial Institute

ABSTRACT Short-time, high-temperature creep properties are reported for several structural aircraft sheet materials. The materials include a titanium alloy, three Alclad aluminum alloys, four low-alloy steels, and two stainless steels. Each group of materials was tested over a useful temperature range. The time of interest for each material ranged from about 1/2 to 120 minutes.

The data obtained are reported as (1) time-deformation curves, (2) design curves including total deformations, (3) stress-total deformation curves which rate the materials after 1, 10, and 60 minutes in test, and (4) creep-design curves. The creep-design curves contain only actual creep deformation in the range of 0.1 to 5 per cent.

This report is Part 3 of a project that was undertaken to obtain the aforementioned data.

WADC TR 52-101 (Part 2)

August 1954

SUBJECT: EQUIPMENT FOR TESTING THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS

INVESTIGATOR: Lawrence A. Shepard, Carl D. Wiseman, C. Dean Starr, John E. Dorn

CONTRACT: AF 33(038)-11502

CONTRACTOR: University of California

ABSTRACT: Four creep testing units were constructed at the University of California to test the creep strength of aircraft metals under intermittent loading and heating conditions. The equipment was designed to permit operations of loading, unloading, heating and cooling of the test specimen to occur automatically and periodically on a preset cycle. Special care was taken to provide smooth and vibration-free function of the equipment. Provisions were made for the accurate and continuous recording of both strains and temperature throughout the test.

WADC TR 54-183

March 1955

SUBJECT: THE INVESTIGATION OF POROUS MEDIA PREPARED FROM SPHERICAL METAL POWDER PARTICLES

INVESTIGATOR: Frank W. Heck, Donald P. Ferriss, Lambert H. Mott, Gregory J. Comstock

CONTRACT: AF 33(616)-396

CONTRACTOR: Stevens Institute of Technology

ABSTRACT: The processing of spherical stainless steel powder particles by compacting and sintering methods is described. Room temperature air permeability and tensile strength and stress rupture data at 1100°F are reported.

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Porous specimens were prepared from six particle size fractions to densities ranging from 75% to 90% of the density of the solid material and the thicknesses ranging from 0.040 inch to 0.200 inch by various combinations of compacting and coining pressures. Sintering time, temperature, and atmosphere were held constant. Methods of separating spherical particles from an aggregate of particle shapes are discussed.

The permeability data are presented in the form of a D'Arcy coefficient of permeability and also as a relationship between the pressure differential across the compact and the volumetric rate of air flow through the compact.

WADC TR 54-598

March 1955

SUBJECT: RETAINER MATERIALS FOR AIRCRAFT GAS TURBINE BEARINGS
INVESTIGATOR: Eugene J. Bucur, F. Clifton Wagner
CONTRACT: AF 33(616)-2099
CONTRACTOR: Horizons, Inc.
ABSTRACT: A study of the elevated temperature wear resistance of potential cage materials for aircraft turbojet bearings was conducted using a special wear testing machine to simulate roughly the conditions to which such bearings are subjected in service. All tests were conducted in the Mechanical Metallurgy Department of Horizons Incorporated.

It has been found that:

1. Several alloy compositions have been developed which have superior bearing properties than "S" Monel and iron-silicon bronze. All of the promising materials except one contain silver as a major alloying element.
2. It has been established that the addition of from 2 to 4% silicon is distinctly beneficial to the wear properties of several classes of metallic alloys.
3. It has been shown that the alloy composition can be varied considerably with respect to the strong, load supporting phase as long as silver is contained in the soft matrix.

WADC TR 55-34

April 1955

SUBJECT: AN INVESTIGATION OF MATERIALS FOR CAGES FOR AIRCRAFT GAS-TURBINE ROLLING-CONTACT BEARINGS
INVESTIGATOR: W.A. Glaeser, C.M. Allen, S.L. Fawcett
CONTRACT: AF 33(616)-2100
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: An investigation has been carried out to develop new materials for cages for rolling-contact bearings used in jet aircraft. Two machines have been constructed to evaluate prospective materials. There are the rubbing-button-test machine and the cage-material testing machine. The rubbing-

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button machine is designed to screen the various materials in the simplest way possible, evaluating their resistance to galling and to seizure under thin-film lubrication at high rubbing velocities. The cage-material testing machine was designed to further evaluate promising materials from the screening tests under conditions simulating actual turbine-bearing operation. Material comparisons are based on the performance of silicon-iron bronze in these machines. At present, this cage material is used more widely than any other material for turbine bearings.

Two promising materials have been developed in the course of this research. These are chromized steel and boron nitride cermet. These materials have exhibited good corrosion resistance to hot MIL-L-7808 products used in the lubrication of turbine bearings.

An initial series of runs using the cage-testing machine with silicon-iron bronze specimens has proved the machine to be a valuable tool for simulating cage-pocket wear by the rolling elements. Comparison of cage specimens from this machine with cages from a bearing operated in a high-speed turbine showed marked similarity in the wear patterns. The loads on the cage pockets were measured with a strain-sensing device.

PACKAGING

WADC TR 53-133 (Part 2)

April 1955

SUBJECT: THEORETICAL INVESTIGATION OF THE MECHANISM OF TRANSFER OF MATERIALS THROUGH POLYETHYLENE
 INVESTIGATOR: Henry A. Bent, Jules Pinsky
 CONTRACT: AF 33(616)-112
 CONTRACTOR: Plax Corp., and University of Connecticut
 ABSTRACT: The temperature variation of the P-factor has been found to be accurately represented by the two parameter equation

$$P = P_0 e^{-E_p/RT}$$

in which E_p and P_0 are constants for the particular permeant polymer system. These parameters are calculated from carefully constructed log vs. $1/T$ plots.

The size, shape, and polarity of the penetrant molecule are factors influencing E_p and for substances no more polar than acetone the expression

$$E_p = 0.0348V + 0.75 V/L + 2.444H$$

reproduces the data to within about 0.5 kcal/mol. where polyethylene is the permeable film.

It is established that for four homologous series that

$$\log P_0 = m \frac{E_p}{RT} + b$$

where m and b are constants peculiar to each series.

Master plots for interpolating homologous materials and extrapolating for temperature changes are included.

WADC TR 53-38 (Part 15)

July 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS
PART 15 THE PERFORMANCE OF VOLATILE CORROSION INHIBITORS
FOR PACKAGING ANTIFRICTION BEARINGS (SERIES N-N)

INVESTIGATOR: A. A. Mohaupt, R.K. Stern

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory

ABSTRACT: An experiment was conducted at the Forest Products Laboratory to evaluate the use of volatile corrosion inhibitors (VCI) for packaging antifriction bearings. Bearings with or without an oil film were sealed in tin cans or flexible, water vaporproof pouches with 1 of 6 VCI materials and subjected to a 3-week cyclic exposure plus 60 days of storage at 120° or 160°F. and 92 percent relative humidity.

Results indicate that some VCI materials can be used advantageously for packaging antifriction bearings.

Under all test conditions, the inhibitor consisting of a kraft paper impregnated with sodium nitrite, urea, and monoethanolamine benzoate gave the best results. A kraft paper impregnated with an amine salt also protected the bearings from corrosion under all conditions, except when used in a pouch exposed to 160°F.

Kraft papers coated or impregnated with VCI compounds produced more desirable results for packaging antifriction bearings than inhibitors in crystalline or powdered form.

In general, the use of an oil film in conjunction with a VCI material did not reduce corrosion under the conditions of this test.

Better results were obtained with certain inhibitors in tin cans than with the same inhibitors in pouch packages.

WADC TR 53-38 (Part 16)

February 1955

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS PART 16. THE EFFECT OF
EXPOSURE ON GLASS PACKS CONTAINING STEEL ROLLERS IN VARIOUS
PRESERVATIVES OR GREASES AND IN ATMOSPHERES OF AIR OR NITROGEN
(SERIES V)

INVESTIGATOR: V. W. Meloche, A. Frisque

CONTRACT: AF 18(600)-103

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CONTRACTOR: Forest Products Laboratory

ABSTRACT: This experiment was conducted primarily to determine the compatibility of various materials used for packaging antifriction bearings under controlled conditions. Test specimens consisted of sealed glass tubes each containing a steel roller, a synthetic grease or petroleum-base preservative with various amounts of moisture, and an atmosphere of air or nitrogen. These specimens were subjected to a 3-week cycle with temperatures ranging from -65°F to 160°F. followed by 60 days of storage at 160°F.

When no water was added to the test specimens, practically no corrosion or stain occurred on the rollers with any of the packaging combinations. When water was added, stain and corrosion were found on the rollers. This test also demonstrated that air or oxygen was a protective passivating agent. In general, the degree of stain and corrosion on rollers in the oxygen containing atmosphere was less than the stain and corrosion on rollers in a nitrogen atmosphere. There was a definite variation in the performance of the various preservatives and greases.

PAINT REMOVER

AF TR 5713 (Sup. 1)

January 1955

SUBJECT: THE DEVELOPMENT AND EVALUATION OF PAINT REMOVER USED BY THE UNITED STATES AIR FORCE

INVESTIGATOR: Sam Collis

ABSTRACT: Paint and lacquer remover formulations containing solvents other than methylene chloride were investigated. Materials evaluated were those submitted by manufacturers, and commonly available solvents and chemicals.

A number of solvents were evaluated for their ability to remove paint and lacquer coatings from metal. Materials for use as surface active agents, activating agents, thickeners, and evaporation retardants were evaluated.

The best experimental formulation developed was nitromethane-toluene-ethanol solvent mixture with a dibutyl amine - monoisopropyl amine-adipic acid thickener, a surface active agent, and a paraffin evaporation retardant. This formulation is considered unsatisfactory for Air Force use due to slow paint removal and poor storage stability.

Many of the materials tested were not developed or intended by the manufacture for the conditions to which they were subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

PETROLEUM PRODUCTS, FUELS

AF TR 5636 (Sup. 1)

December 1954

SUBJECT: THE STORAGE CHARACTERISTICS OF FUELS UNDER SEVERE AND MODERATE CLIMATIC STORAGE CONDITIONS
INVESTIGATOR: Robert W. Altman
ABSTRACT: This program comprised a study of the stability of conventional reciprocating engine type fuels and jet propulsion fuels in an attempt to obtain correlation with the results of desert fuel storage presented in the basic report.

Sixty-one reciprocating engine fuels were aged under moderate outdoor climatic conditions at Wright-Patterson Air Force Base, and eighteen jet engine fuels were subjected to aging in a hot room maintained at $130^{\circ} \pm 2^{\circ}\text{F}$. Subsequent to aging under these conditions for various interim periods, these fuels were tested in the laboratory for their residue content by the ASTM air-jet evaporation method.

A majority of the reciprocating type fuels after 59 months under moderate climatic storage conditions had a gum content above the 6.0 milligrams per 100 milliliters of fuel level which was adopted at the outset of the test as the maximum allowable gum content. At 12 months the jet fuels in hot room storage exhibited such good stability that no further tests were conducted until the 35th month of this storage. At that time seven exceeded the 6.0 milligrams per 100 milliliters of fuel level.

WADC TR 52-35 (Sup. 2)

August 1954

SUBJECT: RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT FUELS
INVESTIGATOR: G. W. Jones, M.G. Zabetakis, G.S. Scott, A.L. Furno
CONTRACT: AF 18(600)-151
CONTRACTOR: United States Department of The Interior
ABSTRACT: The results of limit-of-flammability, limit-of-ignitibility, limit-of-propagation and ignition temperature tests conducted on aircraft fuel and pure hydrocarbon vapor-air mixtures, with various quantities of added nitrogen and carbon dioxide, by the U.S. Bureau of Mines, Gas Explosions Branch between 1 February 1953 and 1 February 1954 are presented. Two aviation gasolines, three jet fuels and six pure hydrocarbon fuel components were used in the investigations.

WADC TR 52-35 (Sup. 3)

February 1955

SUBJECT: RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT FUELS
INVESTIGATOR: G.W. Jones, M.G. Zabetakis, G.S. Scott, A.L. Furno
CONTRACT: AF 18(600)-151
CONTRACTOR: United States Department of The Interior
ABSTRACT: The results of limits of flammability, limit of propagation and

ignition temperature tests conducted on aircraft oil and on aircraft fuel components in air by the U.S. Bureau of Mines, Gas Explosions Branch, between 1 February 1954 and 1 October 1954 are presented. One aircraft oil and a number of pure paraffin and aromatic hydrocarbons were used in the investigations.

WADC TR 53-63 (Sup. 1)

July 1954

SUBJECT: STORAGE STABILITY OF JET TURBINE FUELS
INVESTIGATOR: C.A. Cole, A.C. Nixon
CONTRACT: AF 18(600)-37
CONTRACTOR: Shell Development Company
ABSTRACT: The present phase of the investigation of jet fuel stability has been concerned with the evaluation of practical methods of improving stability through the employment of existing conventional methods of treatment and inhibition. In addition to this, studies to determine the effects of storage variables on gum formation and the influence of aging on such fuel characteristics as filterability and freezing point have been continued.

In the treatment study attention has been focused primarily on gas oil components since previous work has demonstrated that the stability of the final blend is often dominated by the properties of this fraction. The results of this work may be summarized as follows: Relatively severe treatments with sulfuric acid, caustic soda, clay, combinations of the three or mild hydrogenation did not improve the inhibitor susceptibility of any of the fuels tested. Inhibitor response was observed under accelerated aging conditions in the case of full range fuels in which catalytically cracked gas oil made up the sole heavy component. This response, however, became less marked, or negligible, under milder conditions of aging such as in the oven at 70°C, or in the hot room at 110°F.

Considerable improvement in stability was observed in the case of the more unstable cracked blends which had received sulfuric acid, or combinations of treatments including sulfuric acid or hydrogenation. In cases of improved stability, following treatment, significant reductions in maleic anhydride values, mercaptan sulfur, total sulfur and total nitrogen values were observed. Little change in olefin content was obtained following sulfuric acid treatment singly or in combinations of treatments but large reductions in this value were effected by hydrogenation. Hydrogenation appeared superior from the standpoints of stability improvement and fuel loss due to treatment. Treatment improves filterability of ovenaged samples in most cases only in so far as it improves stability with respect to flocculant insoluble gum.

Oxygen availability in desert aging containers has been shown to be important in determining the extent of fuel degradation during storage. The oxygen content of the vapor spaces of drum samples following the summer

of 1952 ranged from 0 to 6% for cracked fuels and 1.5 to 17% for straight run fuels. Samples with fuel to air ratios of 9 have shown much lower gum values than the same fuels aged at 1/1 ratios. The JP-4 fuels have a greater inherent instability than their JP-3 equivalents due to their lower vapor pressure and the consequent greater partial pressure of oxygen in the vapor space of the storage containers. Although fuel insoluble gum levels have shown changes during the last year of desert storage from a decrease to an increase, the filter clogging tendencies of the fuels have in most cases increased. Decreases in MAV values greater than 2 meq/liter with these fuels have usually been accompanied by significant increases in the quantities of soluble and insoluble gum formed. Several inhibitors which appeared promising in the laboratory have not been effective in reducing gum formation on further evaluation in desert storage tests.

Supplemental data on the effect of fuel aging on dielectric constant have confirmed earlier work and show that neither aging time nor soluble or insoluble gum has a significant effect on this value.

WADC TR 53-63 (Part 2)

February 1955

SUBJECT: STABILITY OF JET (TURBINE) FUELS IN STORAGE
INVESTIGATOR: Charles A. Cole, Harry B. Minor, Alan C. Nixon, Thurston Skei, Roy E. Thorpe
CONTRACT: AF 18(600)-37
CONTRACTOR: Shell Development Company
ABSTRACT: Various methods for improving jet fuel stability have been investigated at the Emeryville Research Center of Shell Development Company. Mild hydrogenation of cracked gas oil components appears capable of increasing fuel availability. No effective gum inhibitors have yet been found for desert storage conditions. The effects of other factors on desert storage are summarized. Electron microscopic examination showed filterability to be related to the type of insoluble material formed in aged fuels. The effects of inhibitors, dispersants and fuels were studied in a laboratory test which measured filter clogging tendencies under conditions simulating those in aircraft fuel-oil heat exchangers. Fractionation by distillation and chromatography of a catalytically cracked gas oil into its type components showed that stability under mild aging conditions generally decreased with increasing boiling point and with increasing olefinicity. For this phase of the program, a chromatographic method (chromatagum) was developed for determining gum in small samples.

This investigation is continuing.

WADC TR 54-328

September 1954

SUBJECT: CHROMATOGRAPHIC DETERMINATION OF GUM IN FUELS
INVESTIGATOR: A. C. Nixon, T. Skei, H. S. Knight
CONTRACT: AF 18(600)-37
CONTRACTOR: Shell Development Company
ABSTRACT: A chromatographic method of determining gum in small samples has been developed in order to solve problems in the investigation of jet fuel stability

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being carried out for the Air Force. Although the method was primarily intended for use with experimental fuels and components of limited availability, the simplicity, rapidity and ease of application may make it attractive under other circumstances.

The present introductory report deals with the use of this method with jet and diesel fuels and light fuel oils where its agreement with current procedure is adequate. Preliminary data suggest it may also be applicable to gasolines. Further investigation of the method is in progress to establish optimum procedures.

WADC TR 54-328 (Part 2)

March 1955

SUBJECT: CHROMATOGRAPHIC DETERMINATION OF GUM IN FUELS
INVESTIGATOR: H. S. Knight, T. Skei, A. C. Nixon, S. Groennings
CONTRACT: AF 18(600)-37
CONTRACTOR: Shell Development Company

ABSTRACT: A novel method for the determination of soluble gum in jet fuel, gas oil and possibly also in gasoline was introduced in a previous report. The gum content is related to the length of a brown zone observed when the fuel is displaced over silica gel in a small chromatographic column with α -methylnaphthalene as solvent and acetone as eluent. This method is superior to the conventional procedure involving evaporation at elevated temperatures in a jet of steam because it is much simpler and permits the use of very small samples which is advantageous in research work on fuel stability.

The present, supplementary report deals with improvements of this "chromatogum" method. The purification of α -methylnaphthalene has been made more effective, the necessity of using a closely specified amount of sample has been eliminated, and a narrower column has been designed to improve the reliability even though smaller samples are employed. Also, the method appears to be applicable to insoluble gum from jet fuels and many gas oils.

WADC TR 54-596

May 1955

SUBJECT: SPECTROPHOTOMETRIC - CUPRETHOL METHOD FOR THE QUANTITATIVE
DETERMINATION OF COPPER IN AVIATION FUELS
INVESTIGATOR: O. M. Ballentine

PETROLEUM PRODUCTS, HYDRAULIC FLUIDS

WADC TR 54-191

December 1954

SUBJECT: THE DEVELOPMENT OF A HIGH TEMPERATURE AIRCRAFT
HYDRAULIC FLUID
INVESTIGATOR: N. W. Furby, R. O. Bolt, R. L. Peeler, J. M. Stokely
CONTRACT: AF 33(038)-9831
CONTRACTOR: Standard Oil Company of California

ABSTRACT: The requirements for a high temperature hydraulic fluid possessing favorable viscosity, volatility, lubricity and thermal stability led to the investigation of silicon containing compounds as possible new base materials to meet this demand. The studies conducted by the California Research Corporation have resulted in the synthesis of a number of siloxanes and disiloxanes in an

effort to develop a finished fluid that would meet the requirements set forth in the program. Data are presented on compounds synthesized and on evaluation of fluid blends.

WADC TR 54-532

March 1955

SUBJECT: HIGH TEMPERATURE HYDRAULIC FLUIDS
INVESTIGATOR: Edward S. Blake, James W. Edwards, William C. Hammann
CONTRACT: AF 33(616)-2623
CONTRACTOR: Monsanto Chemical Company
ABSTRACT: An extensive literature search and thermal stability screening of available chemicals were conducted as a basis for planning a research program for the development of a high-temperature (700°F) hydraulic fluid. Esters of organic and inorganic acids offer the best possibilities from the standpoint of ease of synthesis, fluidity and viscosity. Phenolic esters have better thermal stability but poorer hydrolytic and viscosity properties than esters of aliphatic alcohols. Research effort therefore should be directed toward the improvement of the thermal stability of the aliphatic esters and toward the improvement of the viscosity and hydrolytic stability of the aromatic esters. Suggestions for a research program have been made and the thermal stability screening test data are reported for 42 compounds.

WADC TR 55-62

March 1955

SUBJECT: A SONIC SHEAR METHOD FOR DETERMINATION OF SHEAR BREAKDOWN ON HYDRAULIC FLUIDS AND LUBRICATING OILS
INVESTIGATOR: A. J. Girona, Earl B. Essing, Bernard Rubin
ABSTRACT: A procedure has been devised which, within short test periods, will evaluate the shear stability of Hydraulic Fluids and Lubricating Oils containing polymeric materials. This procedure utilizes the Raytheon, 10KC, Magnetostrictive Oscillator Model DF-101. The test fluids are subjected to sonic vibration for a specified length of time, after which viscosity measurements are taken in order to determine the extent of molecular decomposition. Data of typical shear-time relationship of several hydraulic fluids are presented.

PETROLEUM PRODUCTS, LUBRICANTS

WADC TR 53-83 (Part 2)

March 1955

SUBJECT: DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASES
INVESTIGATOR: Edward A. Swakon, Cecil G. Brannen
CONTRACT: AF 33(038)-23687
CONTRACTOR: Standard Oil Company (Indiana)
ABSTRACT: In the work directed toward the development of an aircraft grease suitable for use over the temperature range of -65° to 450°F, emphasis has been placed on the development of thickeners for silicone fluid and on the evaluation of greases at high temperatures. Studies were made on silicone greases thickened with substituted ureas and other materials which might be expected to produce thermally stable greases. A new class of non-soap, organic thickening agents, called arylureas, has been developed. Greases made with arylureas are extremely heat stable, are easily made in conventional grease equipment, exhibit good rheological properties, and have out-performed all other greases in the bearing

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test at 450°F. The lowest temperature at which these greases meet the values given in the requirements is around -40°F. Twenty-six of thirty tests on eleven compositions made with DC-550 Silicone Fluid and arylureas ran between 350 and 1170 hours. On two greases designated MLG-9300 and MLG-9301, ten of fourteen tests ran well over 500 hours. They meet all of the other Air Force requirements except performance in the Navy gear-wear test. Failure in this test is caused by the inherently poor lubrication characteristics of silicone oil and attempts to improve the lubricity by the incorporation of additives have been unsuccessful.

Another arylurea grease made with DC-510 Silicone Fluid closely approached the Air Force requirements. This grease is suitable for use at -90°F. Three bearing tests at 450°F average 270 hours.

Bearing tests on greases comprised of DC-550 Silicone Fluid and the following miscellaneous thickeners-carbon black, copper phthalocyanine, calcium acetate, indigo, and N-benzoyl-p-amino-benzoic acid ran between 20 and 427 hours.

The utility of arylureas is not limited to silicone fluids. Arylurea greases with good rheological properties were made with the following types of fluids: petroleum oils, monoesters, diesters, fluorocarbons, phosphate esters, alkyl silicates and polyalkylene glycols. If high stability of a fluid could be combined with good lubricity, a truly multipurpose grease could be made.

WADC TR 53-293 (Part 5)

March 1955

SUBJECT: HIGH-TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS
PART 5. EVALUATION OF ADDITIVES, SYNTHESIS OF NEW COMPOUNDS,
AND MECHANISM STUDIES

INVESTIGATOR: James W. Cole, Jr., Gordon P. Brown, A. Chandler Schmalz

CONTRACT: AF 33(038)-22947

CONTRACTOR: University of Virginia

ABSTRACT: The oxidation characteristics have been determined of various blends of selected additives with fluids of the di-ester, silicate, silicone and tri-(ortho-chlorophenyl) phosphate types in the absence and presence of aluminum, copper, magnesium, silver, cold-rolled steel and titanium. The temperature range was 200-250°C (400-482°F) with the majority of the runs at 240°C (465°F). A few runs with DC Silicone 550 were conducted at 600° and 700°F. Two experimental methods were employed. One was the previously described standard oxidation-corrosion method in which the fluids were heated under reflux with dry air bubbling through for specified periods of time. The other involves the same test cell, modified for removing small samples of the heated fluid at intervals. Many additives were compared with phenothiazine in di (2-ethylhexyl) sebacate and with phenyl-alpha-naphthylamine in mixed

C₆-C₈ silicates. A number showed promise and recommendations are made for further study. At 400°F, the time needed to bring the oxidizing mixture to a definite break in the viscosity was roughly proportional to the molar equivalent concentrations. As the temperature was raised the effect of the additives became less pronounced until at 482°F the effect was only a slight decrease in the overall rate of oxidation. The behavior of the test metals was not always uniform but in general it was shown that magnesium was most active followed by copper and silver. A spectacular formation of Cu₂Se and Ag₂Se was observed with dilauryl selenide which was otherwise an effective antioxidant. Titanium, aluminum and cold-rolled steel were mostly without well defined effects.

A blend of disiloxane-silicone (MLO 8200 type) was examined to determine its oxidation pattern in the range 400-482°F. Oxidation promoted a decrease of about 30% in viscosity which was not dramatically affected by additives at the upper temperatures. After a short intermediate period of little change, the viscosity of the uninhibited fluid increased rapidly to about 50% of the original in 72 hours at 240°C. The presence of 2% dioctyl diphenylamine held the viscosity unchanged from the initial decrease for about 90 hours after which there was a steady increase in viscosity. Several additives prolonged the intermediate period of little viscosity change, but none was superior to diphenylamine. The silicones were only slowly oxidized at 465°F, but at 600°F the oxidation was rapid; in 24 hours the viscosity increased more than 100%. A few runs with tri-(*o*-chlorophenyl) phosphate at 240°C (465°F) indicated this oil in the absence of inhibitors to have the greatest oxidative stability of any fluid examined under this contract. However, in the presence of copper, oxidation was rapid with considerable reaction with the metal to form cuprous chloride. A mixture of the fluid containing phenothiazine and sebacic acid in the presence of copper oxidized even more rapidly.

Considerable attention was given to showing that the products of thermal and photo-oxidation of phenothiazine are identical and to relating their behavior to possible mechanisms of antioxidant activity. It is postulated that phenothiazine is active as an antioxidant, not so much in itself, as by being oxidized to 3-hydroxyphenothiazine which reduces hydroxyl free radicals to water. The antioxidant is thus converted to a semiquinone or other semi-stable free radical, which can then undergo further oxidation or be reduced back to the antioxidant. The postulated mechanism also affords an explanation of synergistic action and the fact that the same oxidation products are formed during the slow induction period as during the more rapid uninhibited oxidation.

The results of an extensive synthetic program on phenothiazine derivatives are reported. Attention was given to synthesis of derivatives in

the three, four and ten positions and to synthesis of derivatives of 7-benzo (c) phenothiazine. Where sufficient quantities were prepared they were evaluated as antioxidants. Several were highly effective especially the nitrogen substituted alkyl derivatives.

WADC TR 53-337 (Part 2)

February 1955

SUBJECT: POLYNUCLEAR AROMATIC COMPOUNDS FOR HIGH TEMPERATURE LUBRICANTS

INVESTIGATOR: Charles F. Raley, Jr.

CONTRACT: AF 33(616)-276

CONTRACTOR: Southwest Research Institute

ABSTRACT: The properties of triaryl esters of phosphoric acid were studied with regard to their suitability as high-temperature lubricants and related materials. It was found that naphthyl derivatives did not have sufficient thermal stability to compensate for the unwanted increase in viscosity and melting point. The 4-biphenyl group possessed all the advantages of the naphthyl group with much better thermal stability. One particular compound prepared, bis(p-chlorophenyl) 4-biphenyl phosphate had a thermal stability of over 510°C (950°F), by far the highest stability of any compound prepared under this contract. It was found that the chlorophenyl phosphates possessed outstanding thermal stability and seemed to be the most promising type of compound for further development. It was found possible to vary considerably the physical properties of a compound by incorporating three different groups. Finally, the pronounced volatilizing and viscosity-reducing effect of the trifluoromethylphenyl group was observed in several compounds which contained one or more of these groups. It was also found possible to compile a table of approximate thermal decomposition temperatures of numerous aromatic groups.

WADC TR 53-426 (Part 2)

April 1955

SUBJECT: ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS

INVESTIGATOR: Henry Gilman, Richard D. Gorsich

CONTRACT: AF 33(616)94

CONTRACTOR: Iowa State College

ABSTRACT: An extension has been made of the preliminary screening of organo-metallic and organo-metalloidal compounds for thermal stabilities in connection with applications involving high-temperature lubricants and related materials. On the basis of current studies it appears not desirable to give serious consideration to organo-metallic compounds of tin and of lead. Organosilicon compounds of various types are distinctly promising, and the studies now reported and in progress indicate some of the more promising radicals or groups that warrant incorporation in the organo-metallic and organo-metalloidal types. The effects of some of these groups are considered in the discussions which follow the experimental sections.

It is desirable to extend the organogermanium studies, for some of these may enjoy an advantage over corresponding organosilicon compounds in

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a property like higher thermal stability. Interesting properties have been observed with amines and with some organic phosphorous compounds (of the R_3PO type).

In the experimental part there is included a table which outlines the preliminary screening of one hundred and thirty-seven compounds.

WADC TR 54-37

October 1954

SUBJECT: DEVELOPMENT OF A GEAR AND SPLINE LUBRICANT TESTER
INVESTIGATOR: John Morris, Joseph Goldsworthy, John Scott, Charles Sauter
CONTRACT: AF 33(616)-496
CONTRACTOR: Western Gear Works
ABSTRACT: Results of a compilation of lubricants, lubrication, and lubricant tester data include a selected bibliography with abstracts which has been arranged so that references are listed under the subject heading most applicable.

Design of a gear and spline lubricant tester which will accommodate, as test specimens, all the most popularly used types of gears; such as, spur, helical, worm, straight bevel, spiral bevel, and hypoid, as well as splines, is described. The lubricant tester will be capable of test gear speeds up to 30,000 RPM and tooth loads up to 6000 pounds per inch of face width.

The design of the tester was based on the analysis of information from sources listed in the bibliography, gear lubrication experience, and preliminary design studies of possible new simulation, as well as gear type testers.

It is concluded that a universal type, gear lubricant tester should prove advantageous in selecting lubricants for specific applications, as well as placing lubricants into a general classification of usefulness as gear lubricants.

The compilation of information, analysis, and final design of a gear and spline lubricant tester was conducted by Western Gear Works' Research Engineering Group.

WADC TR 54-46

August 1954

SUBJECT: AN INVESTIGATION OF LITHIUM 9/10 HYDROXYSTEARATE GREASES
INVESTIGATOR: Harvey C. Markle, Jr., Herbert Schwenker
ABSTRACT: The melting points of lithium soaps, which were prepared from hydroxystearic acids, were determined. These soaps were combined with synthetic oils to prepare greases which, in turn, were analyzed to determine their physical and chemical properties. Except for water resistance, oil separation, and mechanical stability, the greases made from lithium 9 or 10 hydroxystearate were found to possess properties equal to those of lithium 12 hydroxystearate.

November 1954

SUBJECT: SYNTHETIC LUBRICANTS FOR AIRCRAFT
INVESTIGATOR: Herbert Schwenker, John A. King, James C. Mosteller
ABSTRACT: As military aircraft fly at higher altitudes and speeds, the requirements for lubricants to afford greater resistance to thermal effects, wider liquid or usable temperature ranges, and longer life has and will continue to be an interesting research and development program. Petroleum oils as a source for greases, aircraft hydraulic fluids, engine oils and special purpose lubricants are rapidly being replaced by synthetic materials and will soon be only of historical interest.

Development of a -65° to 450°F grease for use in anti-friction bearings, electronic devices and other types of aircraft equipment is in progress. Emphasis is being placed on improving the wear characteristics of this grease and extending the low temperature limits. In addition, research and development in greases is being devoted to improvement of grease availability and the investigation of oils and thickening agents.

Synthetic lubricants for turbo-prop and turbo-jet applications have been formulated for use at temperature of -65° to greater than 400°F. High gear loadings encountered in some engine and accessory applications have required extensive research and development of suitable anti-wear additives.

The presently available hydraulic fluids offer a maximum usable temperature range of approximately 250°F. Speed, miniaturization of equipment, together with the necessity of operating hydraulic systems near heat producing bodies has increased this temperature range to greater than 400°F. Experimental fluids of the diester and the organosilicon classes have been developed and are being evaluated.

WADC TR 54-339

February 1955

SUBJECT: DETERMINATION OF THE MECHANISM OF THE INCREASE OF VISCOSITY OF ORGANOSILICON COMPOUNDS AT HIGH TEMPERATURES
INVESTIGATOR: O. F. Senn
CONTRACT: AF 33(616)-168
CONTRACTOR: Stanford Research Institute
ABSTRACT: Oxidation of alkoxy- or aryloxysilanes is inevitably accompanied by hydrolysis. Oxidation rates were determined for tetraphenoxysilane, tetra(2-ethylhexoxy)silane, hexa(2-butoxy)disiloxane, and a series of isomeric tetrapentoxysilanes. The presence of 1020 steel, titanium, and copper during oxidation did not produce large changes in rate as compared to the rate for the test material alone.

The hydrolysis of tetraaryloxy- and alkoxy-silanes apparently proceeds through a stepwise degradation. The rate of hydrolysis is affected largely by structure, being very rapid for tetraphenoxysilane, less rapid for tetra(2-ethylhexoxy)silane, and slow for branched tetrapentoxysilanes.

Pyrolysis is probably the least important factor in the degradation of the tetraaryloxy or alkoxysilanes. No significant degradation was observed with tetraphenoxysilane while tetra(2-ethylhexoxy)silane was degraded only slightly by thermal cracking of the 2-ethylhexyl group.

WADC TR 54-417

March 1955

SUBJECT: METHOD OF DETERMINING THERMAL STABILITY OF SYNTHETIC OILS

INVESTIGATOR: O.M. Ballentine

ABSTRACT: An apparatus has been developed for determining thermal stability of synthetic oils. Thermal decomposition temperatures were determined by plotting vapor pressures over a wide temperature range as $\log P$ vs. $\frac{1}{T}$. The point at which the curve deviates from a straight line relationship will be the point at which thermal decomposition occurs. The following advantages are gained through this method of determining thermal decomposition points; the utilization of small size samples (2.0 to 3.0 grams), a wide temperature of operation (up to 1500°F), a high degree of accuracy in the final results, simplicity of operation (requiring one operator), and increased rapidity of operation.

WADC TR 54-418

February 1955

SUBJECT: PROCEDURE FOR DETERMINING VAPOR PRESSURES OF MATERIALS OF LOW VOLATILITY

INVESTIGATOR: O.M. Ballentine

ABSTRACT: An apparatus has been developed for determining absolute vapor pressures of both liquids and solids that exhibit low volatility characteristics. This method has the following advantages: obtaining vapor pressures up to 1000°F, requiring a minimum of operator's time (approximately one (1) hour), relative simplicity and high degree of accuracy in final results. The method employs Knudsen's equation, based on the kinetic theory of gases, in which the weight loss of material per unit time is proportional to the vapor pressure of the material.

WADC TR 54-464

April 1955

SUBJECT: DEVELOPMENT OF SCHEMATIC ANALYTICAL PROCEDURES FOR SYNTHETIC LUBRICANTS AND THEIR ADDITIVES

INVESTIGATOR: J. J. E. Schmidt, F. S. Bonomo

CONTRACT: AF 33(616)2204

CONTRACTOR: Denver Research Institute

ABSTRACT: General properties and methods for the identification, determination, and separation of components of synthetic greases and synthetic lubricants are presented and discussed.

Included in these components are inorganic and organic gelling agents, soap and urea type thickeners, and such additives as antioxidants. A schematic analytical procedure for the separation of grease components is

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presented along with the application of paper chromatographic methods for the identification and separation of antioxidants in greases and synthetic lubricants.

Initial investigations for the separation and identification of dibasic acid esters in synthetic lubricants are presented.

WADC TR 54-576 (Part 1)

February 1955

SUBJECT: EFFECT OF METALS ON LUBRICANTS (PART 1. DESIGN, DEVELOPMENT AND INSTRUMENTATION OF A HIGH TEMPERATURE BATH)

INVESTIGATOR: Vernon A. Lauer

ABSTRACT: This report is the first of a series of reports concerning the effect of metals on the stability of lubricants at elevated temperatures. In order to accomplish this, equipment capable of attaining these temperatures was required.

This report describes the design, development and instrumentation of a high temperature bath capable of attaining and controlling temperatures up to $700^{\circ}\pm 1.0^{\circ}\text{F}$.

WADC TR 54-580

April 1955

SUBJECT: EVALUATION OF ORGANIC FLUORINE COMPOUNDS FOR USE IN MILITARY AIRCRAFT

INVESTIGATOR: Harold Rosenberg, J.C. Mosteller

ABSTRACT: The evaluation of fluorine compounds for use in military aircraft is one phase of a general Air Force program which has been established for the purpose of obtaining materials of unusual properties and capable of advancing the design of new high-speed aircraft and guided missiles. The desirable properties of fluorine-containing organic compounds include wide liquid range, unusual chemical stability, good electrical conduction, desirable heat transfer characteristics and decreased flammability. Fluorine compounds have, accordingly, been studied for use as fire-extinguishing agents, acid-resistant coatings and greases, non-flammable hydraulic fluids, elastomers, electronic equipment and fungicides.

For extinguishing aircraft fires, particularly those involving rocket propellant mixtures, dibromodifluoromethane and bromotrifluoromethane have been shown to be quite effective. Polymers of monochlorotrifluoroethylene and tetrafluoroethylene have been employed successfully as seals, gaskets and in lubricants in aircraft reaction motors and nitric acid-refueling trailers. Various fluorocarbons, especially chlorofluoroheptenes, have been evaluated as "snuffer" and anti-wear additives for hydraulic fluids. Amongst the "fluorelastomeric" materials, the poly-1,1-dihydroperfluoroalkyl acrylates, $[-\text{CH}_2\text{CH}(\text{COOCH}_2\text{R})-]_n$, have shown the most promise from the standpoint of solvent resistance and low-temperature flexibility. Polyfluoroethylene derivatives have found use in electronic components of military aircraft. Recently,

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fluorodinitrobenzenes have been shown to be effective agents for protecting fabrics against fungi attack.

WADC TR 54-614

April 1955

SUBJECT: SYNTHESIS OF HALOTHIOAMIDES
INVESTIGATOR: Boris Weinstein, J. F. O'Brien

WADC TR 55-30 (Part 1)

March 1955

SUBJECT: FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS
INVESTIGATOR: Merrell R. Fenske
CONTRACT: AF 33(038)-18193
CONTRACTOR: The Pennsylvania State University
ABSTRACT: This report describes the current status of development work on Specification MIL-L-7808 oils and turbo-prop lubricants. Fluids of the PRL 3161 and PRL 3313 types are covered in this discussion. A more complete review of the PRL 3161 type of lubricant is given in Wright Air Development Center TR 53-25 entitled "DEVELOPMENT WORK ON LUBRICANTS FOR AIRCRAFT TURBINE ENGINES".

The viscosity-temperature characteristics of synthetic lubricants are important. On the one hand, a low viscosity at low temperatures is desired to enable low temperature starting, while a high viscosity is desired at high temperatures to provide adequate lubrication, particularly to gears. Linear types of polymeric thickening agents with good shear stability are useful in preparing such lubricants.

A single composition can be formulated to meet both Specification MIL-L-7808 turbine engine lubricant and Specification MIL-L-6387 synthetic hydraulic fluid. Five samples of Specification MIL-L-7808 type fluids have been submitted to the Wright Air Development Center by potential commercial suppliers. Data presented in this report indicate that only one of the five commercial fluids meets the viscosity-temperature requirements of the specification.

Lubricants of the PRL 3313 type containing acidic organo-phosphorus compounds have been investigated. Approximately 15 organo-acid-phosphates and -phosphites have been studied in a PRL 3313 type gear lubricant composition. For the most part, these materials are essentially single chemical compounds. The organo-acid-phosphates, and -phosphites, appear to be considerably more effective than the tri-organo-phosphates and -phosphites in improving the wear and lubrication characteristics of synthetic lubricants. These materials have been evaluated in terms of wear and lubrication, oxidation and corrosion, hydrolysis stability, and their foaming tendencies after mild thin film (250°F) oxidation and corrosion test.

Certain acid phosphites appear to be competitive with acid phosphates as lubrication additives and, in addition, show better corrosion characteristics and hydrolysis stability. The acid phosphates and phosphites

appear to be effective as anti-wear lubrication additives in concentrations below 1.0 weight per cent and in many cases, in concentrations of 0.1 weight per cent. The tri-phosphates, on the other hand, are effective in concentrations of 3.0 to 5.0 weight per cent in the extreme-pressure range of lubricity.

The results of a cooperative lubrication testing program with lubricants designated as PRL 3161, PRL 3312, and PRL 3313 show good general agreement. The relative rating of these fluids by this Laboratory is confirmed by the cooperative tests at other laboratories.

A comprehensive study of the oxidation and corrosion characteristics of synthetic turbine engine lubricants has been conducted. Emphasis has been placed on the bulk oil and thin film oxidation and corrosion characteristics at 347°F (175°C.), the corrosion of lead and lead alloys; the storage stability, and the hydrolysis stability of these synthetic lubricants. Synthetic lubricants of the PRL 3313 and PRL 3161 types show good overall oxidation and corrosion stability at 347°F. These tests include the corrosion of the lead-indium coated bearing surfaces of a Pesco gear pump operating at a test temperature of 350°F. In general, it has been found that large differences exist between the corrosion of pure lead and some lead alloys, such as lead-indium. Indications are that small changes in the neutralization number of some PRL 3313 oils on storage are probably related to the catalytic hydrolysis of the ester base stock.

Foaming in certain of the used samples from turbo-prop engines has been noted. After some research, it has been possible to produce a similar foaming behavior in the laboratory by a 250°F. thin film oxidation and corrosion test procedure. Foaming is not a property common to all ester types fluids containing organo-acid-phosphorous compounds as lubrication additives. It is possible to avoid foaming by proper selection of the additives.

Used samples of PRL 3161 and PRL 3313 fluids from turbo-prop engines have been evaluated in terms of viscosity stability, foaming characteristics, wear properties, and oxidation and corrosion stability. With the exception of the foaming properties of used PRL 3313 fluid, all of these lubricants show good service stability.

Analysis of a deposit from the combustion chamber of a turbo-prop engine has been made by this Laboratory. This deposit resulted from the leakage of PRL 3313 fluid into the combustion chamber through a faulty oil seal. There is no evidence that the polymeric thickener, oxidation inhibitor, or phosphate lubrication additives are concentrated in this deposit. The deposit does not differ from that expected from the synthetic ester base stock.

Viscosity-temperature characteristics and oxidation and corrosion stabilities have been evaluated for improved Specification MIL-L-6387 synthetic fluid compositions. The applicability of synthetic fluids

and lubricants of Specification MIL-L-7808 and Specification MIL-L-6387 types to use a crankcase lubricants in aircraft piston engines and hydraulic fluids in certain Navy applications is discussed briefly.

A brief comparison is given of some of the various types of synthetic hydraulic fluids and lubricants now under development by the Services.

A trend toward decreased oxidation and corrosion stability since World War II in Specification MIL-O-5606 and Specification AN-O-9 fluids is indicated.

The status of development work on an ethyldibromobenzene type of non-inflammable hydraulic fluid is reviewed. Laboratory evaluation of this fluid has essentially been completed. Toxicity studies and tests in a hydraulic "mock up" system are now being conducted by the Wright Air Development Center. Limited evaluations have been completed for a phosphate-base fluid and a Silicone-diester-fluoro-carbon fluid.

The quantitative distribution of oxygen in oxidized esters has been measured. A satisfactory oxygen balance has been obtained with these techniques.

Studies have been made on the effect of metal catalysts on the oxidation and corrosion stability of certain phenothiazine-inhibited esters. The effectiveness of phenothiazine in esters and the effect of oxidation and corrosion conditions on the concentration of phenothiazine are reported. A technique is given for measuring quantitatively the concentration of phenothiazine in various esters. There is some indication that diphenyl amine or other materials containing aromatic ring structures may result from the oxidative deterioration of phenothiazine.

A survey has been made of the oxidation and corrosion characteristics at 500°F of several types of esters and other synthetic lubricants. The oxidation stability of di-2-ethylhexyl sebacate at 500°F has been studied in some detail. Little or no induction period has been noted in these tests at 500°F. The variables affecting oxidation rate appear to be quite critical in achieving reproducibility.

Some bulk oil oxidation and corrosion tests have been conducted at 400°F. with conventional phenothiazine-inhibited ester type lubricants. The induction period at 400°F. appears to be about one-eighth the induction period at 347°F. This rate of change in induction period with temperature is in good agreement with similar data for mineral oil compositions inhibited with Paranox 441 and meeting Specification MIL-O-5606.

The thermal stability of various types of synthetic lubricants has been evaluated at 500°F. Thermal stability studies of Acryloid solutions have also been conducted. Studies have been completed on the effect of these

500°F temperatures on the oxidation, corrosion, and shear properties of Acryloid polymers. The effect of mechanical shear, oxidation, and corrosion on thermal stability has also been determined. These results indicate that certain Acryloid polymers are satisfactory for use in high temperature (500°F.) lubricant compositions.

Lubricant studies on esters in the Shell four-ball wear and extreme-pressure lubricant tester are discussed. These studies include the effect of phosphoric acid and acid phosphates as anti-wear additives. The effectiveness of acid phosphates is compared with the tricresyl phosphate susceptibility of various esters. A study of the relative ease of lubrication of titanium and titanium alloys will be conducted in cooperation with Army Ordnance.

Current and future programs are outlined for tests using the Pesco 3000 psi gear pump, the Vickers 3000 psi piston pump, and the revised PRL high shear viscometer. The latter apparatus measures both permanent and temporary viscosity loss.

The current status of new specification tests suggested for inclusion in synthetic lubricant specifications such as Specification MIL-L-7808 and Specification MIL-L-7499 is discussed. These tests include (1) the thin film oxidation and corrosion test, (2) a wear test in the Shell four-ball wear and extreme-pressure lubricant testers, and (3) a vapor pressure and evaporation test.

This Laboratory will likely also carry out a study of thin film rust and corrosion preventives for Navy Ordnance.

A list is given of the low temperature viscosity standards, synthetic lubricants, and synthetic lubricant components that this Laboratory distributed during the past year.

WADC TR 55-30 (Part 2)

March 1955

SUBJECT: FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS
INVESTIGATOR: Merrell R. Fenske, E. Erwin Klaus
CONTRACT: AF 33(038)-18193
CONTRACTOR: The Pennsylvania State University
ABSTRACT: This report describes work carried out on a continuing project to develop improved fluid and lubricant formulations for use by the Air Forces. During the period covered by this report, the emphasis has been placed on the development of synthetic turbo-jet and turbo-prop lubricants. The current program deals primarily with the effect of phosphorous-containing lubricity additives on an ester-base formulation with established over-all properties. The applicability of this type additive for use in mineral oil formulations has been examined.

Equipment has been designed and constructed for constant temperature use at temperatures in the range of 300° to 800°F. The oxidation behavior of various classes of synthetic fluids and hydrocarbons has been explored at temperatures of 300° to 500°F. Thermal stability of many of the fluids has been determined at temperatures up to 750°F.

Small quantities of dibasic acids have been shown to be effective metal deactivators for controlling metal corrosion in dibasic acid ester-base fluids and lubricants.

A survey of several brominated aromatics has been made to determine the applicability of these materials as base stock constituents for non-inflammable fluids.

WADC TR 55-30 (Part 3)

March 1955

SUBJECT: FLUIDS LUBRICANTS, FUELS AND RELATED MATERIALS

INVESTIGATOR: E. Erwin Klaus, Merrell R. Fenske

CONTRACT: AF 33(038)-18193

CONTRACTOR: The Pennsylvania State University

ABSTRACT: This report describes work carried out on a continuing project to develop improved fluids, lubricants, and fuels for use by the Air Force. The current report deals primarily with the development of high temperature hydraulic fluids and jet engine lubricants and the study of dirtiness characteristics of jet fuel at elevated temperatures.

The hydraulic fluid development goal is a fluid suitable for use at 700°F. for a limited time in a sealed hydraulic unit. The properties of high quality examples of the following chemical classes have been measured; silicones, silicate esters, diesters, pentaerythritol esters, silicone-ester blends, mineral oils, synthetic hydrocarbons, polyglycol ethers, chlorinated aromatic hydrocarbons, and aryl phosphates. The properties measured include: viscosities from 0° to +700°F., thermal stabilities from 500° to 750°F., oxidation and corrosion stability at 347° and 500°F., and operation in a hydraulic pump (vane type) at 500° to 600°F.

The jet engine lubricant goal is a fluid suitable for use for extended periods at 500° to 600°F. bulk oil temperature. The same general chemical classes enumerated for hydraulic fluids have been included in the engine oil studies. Particular emphasis in the jet engine oil studies has been placed on lubricity studies and severe oxidation and corrosion evaluations at 500°F.

Jet fuel dirtiness has been studied from two basic approaches. An attempt has been made to remove the dirty components from a fuel by physical separation techniques. Secondly, a laboratory method of reproducing high temperature dirtiness in jet fuels is being studied.

Coupled with the above programs, some miscellaneous investigations of hydraulic fluid and lubricant problems have been conducted at the

at the request of the Services. In addition, viscosity standards, experimental fluid and lubricant formulations, and reports prepared by this Laboratory have been distributed at the request of industry and the Services

WADC TR 55-90

May 1955

SUBJECT: LITERATURE SURVEY OF LOW MOLECULAR WEIGHT POLYNUCLEAR AROMATIC COMPOUNDS
INVESTIGATOR: Charles F. Raley, Jr.
CONTRACT: AF 33(616)-276
CONTRACTOR: Southwest Research Institute
ABSTRACT: A literature search was carried out covering the field of low molecular weight polynuclear aromatic compounds with the object of determining the usefulness of such compounds as high-temperature lubricants. The highest literature boiling point, calculated atmospheric boiling point, melting point, and literature reference are given. Recommendations are made as to the compounds or types of compounds which appear promising as high-temperature lubricants.

PLASTICS, STRUCTURAL

WADC TR 52-183 (Supl 2)

October 1954

SUBJECT: ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 BULLETIN, "PLASTICS FOR AIRCRAFT".
INVESTIGATOR: Donald C. Coleman
CONTRACT: AF 33(038)-51-4326E
CONTRACTOR: Forest Products Laboratory
ABSTRACT: Developments in the program of research in plastics for aircraft conducted by the U.S. Forest Products Laboratory during fiscal year 1954 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Two technical reports issued during the fiscal year are abstracted.

WADC TR 52-5 (Supl 3)

April 1955

SUBJECT: HIGH STRENGTH EPON LAMINATES
INVESTIGATOR: Frank C. Hopper
CONTRACT: AF 33(038)-19587
CONTRACTOR: Shell Development Company
ABSTRACT: This report is designed to present an accurate account of the present status of research on EPON resin laminating with reference to this contract. It describes in detail all work performed during the past year and summarizes previous work dating back to the inception of the contract.

Wet and dry laminating systems are described. Wet systems based on EPON 828 yield laminates with exceptional flexural and compressive strengths at room temperature. Resistance to water and solvents is excellent.

Strength retention at elevated temperatures up to 300°F is good.

Dry laminating systems offer good strength from room temperature to 500°F. Laminates made from EPON X-12100 cured with Curing Agents E or F retain up to 36,000 psi flexural strength at 500°F after aging 200 hours at 500°F. Even stronger after short exposure to 500°F, but less resistant to aging at that temperature, is a mixture of EPON 1001 with Plyophen 5023 (a phenolic resin) cured with dicyandiamide.

WADC TR 53-185 (Pt. 2)

May 1955

SUBJECT: A STUDY OF THE RAIN EROSION OF PLASTICS AND METALS
INVESTIGATOR: Roy R. Lapp, Raymond H. Stutzman, Norman E. Wahl
CONTRACT: AF 33(600)-6469
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.
ABSTRACT: Many engineering problems have been encountered with the advent of subsonic and supersonic aircraft. One of these problems is the phenomena of erosion, during flight through rain, of coatings, structural plastic and metal parts on the exterior of high speed aircraft.

This report is a compilation of test data obtained, mostly at 500 mph and 1 inch per hour rainfall, on various metallic and non-metallic aircraft materials, using the rotating arm erosion apparatus.

Flight tests in rain on a jet aircraft were carried out to obtain actual service test data. Air foil shaped specimens of various coatings and plastics were attached to the wing leading edge, and preliminary flights through rain at 500 mph made. These flight tests corroborated laboratory tests and the fact that neoprene coatings such as Gaco N-79 and Goodyear 23-56 possess the greatest erosion resistance of non-metallic materials tested to date.

WADC TR 53-192 (Part 5)

March 1955

SUBJECT: MECHANISM OF RAIN EROSION PART 5. FURTHER STUDIES ON CAVITATION IN LIQUID DROPS ON IMPACT
INVESTIGATOR: Olive G. Engel
CONTRACT: AF 33(616)53-9
CONTRACTOR: National Bureau of Standards
ABSTRACT: The search for evidence of cavitation in the flow of a waterdrop after it impinges against a glass plate at approximately its terminal velocity in air was continued. The schlieren arrangement discussed in the preceding report was used. The interpretation of pictures obtained with this method is discussed, and the evidence of bubble formation which was found in waterdrops saturated with argon gas, waterdrops saturated with

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carbon dioxide gas, and in drops of hydrant water is presented. An equation for the time dependence of the radius of the radial flow of the drop after collision with the glass plate is developed. The time dependence of the velocity of the radial flow is considered. Results of a measurement of the impact force are given.

WADC TR 53-371

October 1954

SUBJECT: HEAT RESISTANT POLYESTER LAMINATING RESINS
INVESTIGATOR: Marvin Botwick, William Cummings, Paul M. Elliott
CONTRACT: AF 33(600)-16825
CONTRACTOR: United States Rubber Company
ABSTRACT: The heat resistance of Vibrin X-1047 laminates is notably influenced by the finish on the glass cloth employed. The heat resistance of such laminates is a more searching test of cloth quality than the measurement of green flexural strengths. A 301-finish is generally satisfactory for use with heat resistant polyesters.

Two monomers, triallyl aconitate and diallyl Δ^4 -3, 6-methanotetrahydrophthalate, in combination with Alkyd X-1038 provide two new resins which show good retention of flexural strength at 300-400°F. With the triallyl aconitate resin, a flexural strength of 27,000 psi is maintained through 408 hours at 300°F. At 400°F the flexural strength is still 26,500 psi after 408 hours exposure. The resin derived from diallyl Δ^4 -3,6-methanotetrahydrophthalate is still increasing in strength between 192 and 408 hours at 300°F (38,900 to 42,100 psi) and does not lose strength in the same interval at 400°F (38,000 to psi level.).

It has been demonstrated that a cured foam of 10 lbs./cu. ft. density can be prepared from Vibrin X-1047 using the technique developed under Bu. Ships Contract Nobs-54183 (1773).

A modified poly (bismethylolxylene maleate) alkyd is superior to the modified poly (ethylene maleate) used in Vibrin X-1047 where resistance to long term exposure to 500°F is involved. Using a mixed triallyl cyanurate - diallyl Δ^4 -3,6-methanotetrahydrophthalate monomer system, rather than triallyl cyanurate alone, affords a superior heat resistant polyester. At the same time the materials cost of the resin is reduced.

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May 1955

SUBJECT: HEAT RESISTANT POLYESTER LAMINATING RESINS
INVESTIGATOR: William Cummings, Fred J. Foster, Robert G. Nelb
CONTRACT: AF 33(600)-16825
CONTRACTOR: United States Rubber Company
ABSTRACT: Diallyl carbate in combination with triallyl cyanurate and VIBRIN 135 alkyd provides a new resin with improved retention of flexural strength after aging 192 hours at 500°F. This new resin has been coded

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VIBRIN X-1068. VIBRIN X-1068 glass fabric laminates exhibit flexural strengths of 26,000 - 30,000 psi at 500°F. after being aged for 192 hours at 500°F. The finish on the glass fabric plays a very significant role in the hot strength of the laminates. The 301 and Garan finishes have given the best results. VIBRIN X-1068 laminates exhibit improved craze resistance over laminates prepared from the conventional triallyl cyanurate based heat resistant polyester resins.

WADC TR 53-483

March 1955

SUBJECT: EFFECTS OF FABRIC FINISH AND WET EXPOSURE ON STRENGTH PROPERTIES OF GLASS-CLOTH POLYESTER LAMINATES
INVESTIGATOR: Fred Werren
CONTRACT: AF 33(038)51-4066
AF 33(038)51-4326
CONTRACTOR: Forest Products Laboratory
ABSTRACT: Strength tests were made of polyester laminates reinforced with 181 glass fabric, having 114 finish, a methacrylic chrome complex, and three of the newer fabric finishes. The tests were made after normal conditioning and after varied exposure conditions, including immersion in boiling water, immersion in water at room temperature, and conditioning in an atmosphere at 100°F and near 100% relative humidity.

This report presents the results of tests made to determine the effect of various exposure conditions, including long-time exposure in a humid atmosphere, on the strength properties of the various laminates. The tests show that the laminates made of fabric with the newer finishes were consistently higher in flexural properties after wet exposure than the 114 finish.

WADC TR 54-326

March 1955

SUBJECT: DETERMINATION OF RESIN CONTENT OF GLASS FIBER POLYESTER LAMINATES CONTAINING A CALCIUM CARBONATE FILLER
INVESTIGATOR: S.D. Toner
CONTRACT: AF 33(038)51-4060
CONTRACTOR: National Bureau of Standards
ABSTRACT: The presence of calcium carbonate filler in unoriented glass-fiber reinforced polyester laminates introduces appreciable errors when determining the resin content by the accepted method.

The temperature range, 538° to 593°C, required to burn off the resin is higher than that at which calcium carbonate begins to dissociate. The reduction of calcium carbonate to calcium oxide and the subsequent evolution of carbon dioxide results in a greater loss in weight than would be expected if no filler were present. A quantitative method of restoring the carbon dioxide to the reduced filler consists of treating the residue with an excess of a concentrated ammonium carbonate solution to convert the calcium oxide formed back to calcium carbonate.

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Specimens were prepared from predetermined amounts of resin, glass-fiber mat and calcium carbonate to give the following composition, by weight: resin, 50 percent; glass fiber, 37.5 percent; and calcium carbonate, 12.5 percent.

The specimens were ignited to constant weight in a muffle furnace, cooled, and weighed. The residue was treated with ammonium carbonate and the increase in weight due to absorbed carbon dioxide was determined. Hot 3N hydrochloric acid was added to the treated residue in an attempt to remove, quantitatively, the calcium carbonate.

Results obtained in these experiments indicate that treatment of the residue with ammonium carbonate will reduce the error in the resin content from an average 3.75 percent to less than 0.1 percent. The acid treatment cannot be used to give quantitative determination of the calcium carbonate, the resin of the glass since about 10 percent of the glass dissolves in this treatment.

WADC TR 54-474 (Part 1)

March 1955

SUBJECT: EVALUATION AND DEVELOPMENT OF PLASTIC LAMINATED BACKING BOARD MATERIALS
INVESTIGATOR: Wendell H. Smith, Charles C. Surland
CONTRACT: AF 33(616)-484
CONTRACTOR: The United States Rubber Company
ABSTRACT: New plastic laminate backing board constructions produced with variations of resins, fabrics, fillers, and fabricating procedures were evaluated and compared with a representative group of commercially available backing board materials.

Test devices were developed to determine biaxial strength properties and resistance to fluid ram action and augment the information obtained by conventional mechanical property tests and cube gunfire tests.

New backing board constructions were produced. Test work and development of methods and equipment were carried out, as well as gunfire and cube gunfire tests.

An improved backing board construction was developed using widely spaced rovings of glass fiber bundles impregnated with polyester resin.

Further work on extensions to this contract will permit more complete investigation of variations in the improved backing board construction, and the use of the developed test methods should provide better screening of improvements.

May 1955

SUBJECT: EVALUATION OF NON-WOVEN GLASS FIBER REINFORCEMENTS FOR LOW PRESSURE MOLDED POLYESTER RESINS FOR AIRCRAFT APPLICATIONS

INVESTIGATOR: F.W. Reinhart, M. F. Toompas, M. C. Slone, I. Wolock

CONTRACT: AF 33(038)-51-4060

CONTRACTOR: National Bureau of Standards

ABSTRACT: The strength properties of several glass-fiber reinforced polyester moldings and sheets made from similar materials were determined. The constructions tested were: tote boxes made from glass mat, and from glass fiber roving preforms with no filler, and with filler; flat sheets made from the same combinations of resins, glass fiber and filler as used in the tote boxes; window frames made from glass fiber roving preforms; and small panels made from glass fiber reinforced molding compound. The results indicate that the bottoms of the tote boxes made from preforms with no filler had slightly higher strength properties than the corresponding sheets. The bottoms of the tote boxes made from glass mat had strength properties approximately equal to the corresponding sheets, and the bottoms made from preforms with filler had lower strength properties than the corresponding sheets. The strength properties of the bottoms of the window frames were approximately equivalent to those of the corresponding tote boxes with no filler and were, in general, higher than those of the corresponding flat sheets. The sides of the tote boxes and the flanges of the window frames had lower strength properties than corresponding construction in the flat sheets. The panels made from the glass fiber reinforced molding compound had much lower strength properties than the other moldings tested. In general, the strength properties of the moldings decreased after immersion in water.

WADC TR 55-31

March 1955

SUBJECT: CURING OF VOID-FREE GLASS-CLOTH-REINFORCED LAMINATES AT ROOM TEMPERATURE

INVESTIGATOR: Bruce G. Heebink

CONTRACT: AF 33(038)51-4326E

CONTRACTOR: Forest Products Laboratory

ABSTRACT: There is a demand for polyester-resin systems that cure at room temperature for use with glass-fiber reinforcing for such applications as prototype aircraft parts or for repair of radomes. Many oxygen-liberating catalysts have been found that will promote cure at room temperature, but care must be exercised in their choice and proportions to avoid too rapid or too slow curing conditions.

Void-free test laminates were made of 181 glass cloth that yielded acceptable strength properties when using one of these room-temperature curing combinations with a typical polyester resin.

An unusual small-void formation developed with certain specific rolls of cloth, which was thought to be due to a chemical reaction between the catalysts and the finish on the cloth.

May 1955

SUBJECT: DIMENSIONAL STABILITY OF PLASTIC MOLDING COMPOUNDS
INVESTIGATOR: B.G. Heebink, W.G. Youngquist
CONTRACT: D.O. (33-616)-53-20
CONTRACTOR: Forest Products Laboratory
ABSTRACT: Cellulose filled melamine molding compounds have been replaced for use as insert insulating material for aircraft multipin connectors by mineral filled polyester compounds with satisfactory results, because of the greater dimensional stability of the latter. The unsatisfactory materials changed in dimensions after being in service for a long period of time to such an extent that the connectors could no longer be connected. This report covers quantitative tests for dimensional stability on a number of different molding compounds of the following types:

1. Cellulose filled melamine
2. Mineral filled alkyd
3. Mineral filled diallyl phthalate

Both high temperature dry condition tests and high temperature high humidity tests were run. The results of the high temperature dry tests show that the cellulose filled melamine compounds all had to from 10 to 40 times the dimensional changes of the other two types. There was not the same clear cut difference in the high temperature high humidity tests. Therefore, the high temperature dry condition test is a more effective screening test.

POLYMERS

WADC TR 54-102

July 1954

SUBJECT: ORGANOSILOXANE POLYMERS CONTAINING POLAR GROUPS IN THE SIDE CHAINS
INVESTIGATOR: Sanders D. Rosenberg, Eugene G. Rochow
CONTRACT: AF 33(616)-479
CONTRACTOR: Harvard University
ABSTRACT: In accordance with the proposed objectives of this research a study of the possible synthesis of nitrogen and boron containing polymerizable silicon monomers of the $p\text{-R}_2\text{MC}_6\text{H}_4\text{Si}(\text{CH}_3)\text{X}_2$, (where M is either nitrogen and boron and X is a hydrolyzable group like chloro or alkoxy) was undertaken. Two nitrogen-containing silicon monomers methyl- p -dimethylaminophenyldichlorosilane and methyl- p -dimethylaminophenyldimethoxysilane were prepared by reacting p -dimethylaminophenyllithium with methyltrichloro- and methyltrimethoxysilane, respectively.

Various methods were attempted to prepare a suitable boron-containing silicon monomer without success. Incidental with these attempts it

was found that p-bromophenyldichloroboron could be prepared by disproportionation of tri-p-bromophenylboron and boron trichloride. Symmetrical organoboron compounds were prepared by reacting the suitable Grignard reagent with boron trichloride or trimethyl borate. Di-n-butyl- and di-n-propylbromoboron were prepared by reacting the respective symmetrical compounds with bromine.

It was possible to prepare Grignard reagents of methyl-p-bromophenyldiphenoxysilane and methyl-p-bromophenyldi-o-cresoxysilane. Only the latter reagent proved to be of use in subsequent reactions.

Trimethylallylsilane and trichlorosilane were condensed, under the activation of benzoyl peroxide, to form trimethyl-3-trichlorosilylpropylsilane. Under similar conditions, trimethylallylsilane did not condense with methyldichlorosilane. It is planned to attempt to condense trichlorosilane with triallylboron and di-p-propylallylboron.

Reference has been made to the difficulties involved in the analysis of organosilicon and organoboron compounds, and some suggestions have been made. From the infrared spectra of the organoboron compounds that were prepared a tentative assignment of the carbon-boron absorption frequency has been made at 1370-1390 cm⁻¹ or 1320-1340 cm⁻¹. Three tables containing the new compounds prepared during the course of this research are included.

WADC TR 54-264

December 1954

SUBJECT: SYNTHESIS OF MONOMERIC MATERIALS
INVESTIGATOR: B. David Halpern, Wolf Karo, Leonard Laskin, Philip Levine, Jack Zomlefer
CONTRACT: AF 33(616)-252
CONTRACTOR: Monomer-Polymer, Inc.
ABSTRACT: In the course of this project, the possibility of preparing the following materials was investigated:

Vinylsilanes
Allyl Sulfides
Divinyl Sulfide
2-Methylthioethyl Vinyl Sulfide
Vinylidene Cyanide
N-(2,2,2-Trifluoroethyl)-acrylamide
N-Alkyl-N-1,1-Dihydroheptafluorobutylacrylamides
N-Alkyl-N-1,1-Dihydroheptafluorobutylmethacrylamides
Methylene Silanes
1,1-Dihydroheptafluoro-1-butyl alpha-(Trifluoromethyl)-acrylate
1,3-Dicyano-1,3-butadiene
Heptafluoropropylacrylamide

Prior to the attempted preparation of monomers, a comprehensive literative survey was undertaken.

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The modification of Acrylon rubbers was investigated by use of both copolymer and terpolymer systems. A preliminary evaluation of the more promising materials was performed.

Samples of monomers and intermediates have been submitted to the Wright Air Development Center.

WADC TR 54-525

February 1955

SUBJECT: RESEARCH ON HEAT-RESISTANT ACRYLATE-SILOXANE ELASTOMERS
INVESTIGATOR: Robert P. Cox, Ralph W. Buetow, Luther L. Yaeger
CONTRACT: AF 33(616)-2135
CONTRACTOR: BJORKSTEN RESEARCH LABORATORIES, INC.
ABSTRACT: The objective of this project was the synthesis of acrylate-siloxane elastomers with good thermal and solvent resistance. Of the many polymers investigated as gum rubber bases, vinyl triethoxy-silane-acrylonitrile-ethyl acrylate compositions were the best as measured by (1) swelling in 70-30 iso-octane-toluene and in Penola oil and (2) weight loss and flexibility after heating in air at 350°F.

The addition of either dibutyl phosphite or polychlorotrifluoro ethylene powder (Kel F) to these gum rubbers generally maintained their flexibility in air at 350°F. for 250 to 1000 hours. As a reinforcing filler to provide good tensile strength, carbon black (Philblack O) was superior to other fillers tested. Trimene Base was effective as a vulcanization accelerator throughout this work.

The vinyl triethoxysilane-ethyl acrylate gum rubbers could be vulcanized without additives through the formation of cross-linkages. Cross-linking probably took place through adjacent SiOH groups formed in emulsion polymerization.

RUBBER

WADC TR 52-191 (Part 3)

December 1954

SUBJECT: FLOURINE CONTAINING ELASTOMERS
INVESTIGATOR: Earl T. McBee, Carleton W. Roberts
CONTRACT: AF 33(038)-20581
CONTRACTOR: Purdue University
ABSTRACT: 1. An improved stepwise synthesis of bis(3,3,4,4,5,5,5-heptafluoropentyl)diethoxysilane has been developed to give the product in overall yield of 55 per cent based on the 3,3,4,4,5,5,5-heptafluoropentylmagnesium bromide.
2. The one step synthesis of bis(3,3,4,4,5,5,5-heptafluoropentyl)diethoxysilane from 3,3,4,4,5,5,5-heptafluoropentylmagnesium bromide and diethoxydichlorosilane gave less than 48 per cent of crude product.

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3. 3,3,4,4,5,5,5-Heptafluoropentylmethyldiethoxysilane was prepared by the reaction of 3,3,4,4,5,5,5-heptafluoropentylmagnesium bromide with methyltriethoxysilane and by the reaction of methylmagnesium bromide with 3,3,4,4,5,5,5-heptafluoropentyltriethoxysilane.

4. 3,3,4,4,5,5,5-Heptafluoro-1-bromopentane was obtained in 39 per cent overall yield from heptafluorobutyraldehyde.

5. Three synthetic routes were investigated for the synthesis of 3,3,3-trifluoropropyl halides, the hydrobromination of 3,3,3-trifluoropropene, chlorination of 1,1,1-trifluoropropane obtained by catalytic reduction of 3,3,3-trifluoro-1,1,2-trichloropropane, and the best method, the liquid phase chlorination of 1,1,1-trifluoropropane from fluorination of 1,1-dichloro-1-propene. The latter method gave the desired product in an overall yield of 42 per cent.

6. Both 3,3,3-trifluoropropylmagnesium bromide and chloride were converted in a stepwise manner in bis(3,3,3-trifluoropropyl)dimethoxysilane; the best overall yield based on 3,3,3-trifluoropropyl halide was 57 per cent.

7. 3,3,3-Trifluoropropylmethyldimethoxysilane was prepared in 62 per cent yield.

8. Several attempts were made to obtain evidence for the formation of a Grignard reagent or a lithium compound from 1,1,1,2,2,3,3,-heptafluoro-4-iodopentane; these were unsuccessful.

9. Attempts to prepare 2,2,3,3,4,4,4,-heptafluoro-1-methylbutyl silanes were unsuccessful.

10. Synthetic steps leading to 2-methyl-3,3,4,4,5,5,5-heptafluoro-2-iodopentane were stopped at the p-toluenesulfonyl ester of the corresponding alcohol.

11. Extensive studies have been completed on the synthesis of perfluoropropylsilanes. The exchange reactions between p rfluoropropyl iodide and methylolithium and perfluoropropyl iodide and phenylmagnesium bromide have been used to prepare perfluoropropyllithium and perfluoropropylmagnesium bromide. These organo-metallics have been caused to react in ether solvents at varying concentrations and temperatures with silanes containing replaceable halogen or alkoxy groups.

12. Perfluoropropylsilanes have been successfully prepared using perfluoropropyllithium and several silanes. Perfluoropropylmethylethoxychlorosilane has been prepared in 10 per cent yield.

13. The decomposition of perfluoropropyllithium to give high percentages of perfluoropropene, the azeotroping tendency of the silanes, and the wide spectrum of products obtained from the reactions of perfluoropropyllithium with silanes has made isolation of desired products a demanding research undertaking.

WADC TR 52-197 (Part 4)

January 1955

SUBJECT: SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS
INVESTIGATOR: F. A. Bovey
CONTRACT: AF 33(038)-515
CONTRACTOR: Minnesota Mining and Manufacturing Company
ABSTRACT: This report describes the preparation and properties of fluorine-containing rubbers. The object of the work is the development of elastomeric materials which are resistant to the fuels, lubricants, and hydraulic fluids used in military aircraft and which are serviceable over the widest possible temperature range.

Of the materials under development, the following appear to be of chief interest:

- A. Perfluorobutadiene Copolymers. Copolymers of perfluorobutadiene with 1, 1-dihydroperfluoroalkyl vinyl ethers offer promising high temperature resistance, low swelling in aircraft fluids, and high resistance to ozone. Difficulties have been experienced in preparing products of consistent quality, and ineffectively vulcanizing and stabilizing the copolymers.
- B. Fluoroacrylates. In addition to the 1, 1-dihydroperfluoroalkyl acrylates, which have been described in earlier reports and which have outstanding solvent resistance but limited low temperature flexibility, newer classes of fluoroacrylates have been synthesized. Of particular interest are the γ -(perfluoroalkoxy)-1, 1-dihydroperfluoropropyl acrylates, which offer solvent resistance at least equal to that of the earlier series but are flexible at temperatures 25°C. lower than the polymers of the 1, 1-dihydroperfluoroalkyl acrylates.

The mechanism of the polyamine vulcanization reaction has been shown to be amide formation. Extensive testing of vulcanizates of poly-1,1-dihydroperfluorobutyl acrylate (poly-FBA) prepared in poly-amine curing recipes has indicated excellent resistance to a variety of liquids at high temperature.

A thorough study of the molecular weight and fundamental solution properties of poly-FBA has been carried out.

November 1954

SUBJECT: PLASTICIZERS FOR OIL-RESISTANT RUBBER
INVESTIGATOR: Charles S. Imig
CONTRACT: AF 33(616)-59
CONTRACTOR: Phillips Petroleum Company
ABSTRACT: Various compounds either purchased or synthesized were evaluated as low temperature, non-extractable plasticizers for oil-resistant polymers such as Paracril B. These compounds including various carbamates, dithiocarbamates, amides, imides, esters, and several liquid polymers were generally unsatisfactory as low temperature plasticizers for Paracril B. While several of these compounds, especially the polymers, were non-extractable in 70/30 isooctane-toluene and others, particularly N,N-dibutylformamide and N,N-dibutylacetamide, imparted good low temperature properties, none was found which combined the desired characteristics of being non-extractable and imparting good low temperature properties.

Attempts to improve the low temperature properties of 70/30 butadieneacrylonitrile copolymer by the addition of ethyl, butyl or hexyl mercaptans was unsuccessful. It appeared that higher mercaptans might improve the low temperature properties but only at some sacrifice in oil resistance.

It was found that a mixture of Thiokol Corporation's liquid polysulfide ZL-109 and TP-90B is an effective plasticizer for Paracril B and imparts fair low temperature properties combined with reduced extractability. However, with stocks containing ZL-109, the scorch time is reduced, the stress-strain properties are impaired, and the cure is retarded. Compounding studies were made to circumvent these deficiencies.

WADC TR 53-372

November 1954

SUBJECT: HEAT-RESISTANT AIR HOSE, HEAT-RESISTANT FUEL AND OIL HOSE, AND HIGH-PRESSURE FUEL HOSE
INVESTIGATOR: Richard J. Meisinger, Theodore D. Ernst
CONTRACT: AF 33(038)-22905
CONTRACTOR: United States Rubber Company
ABSTRACT: Two hoses, a braided type and a fabric type, were developed for use with compressor bleed air at an ambient temperature of 500°F. These hoses are designed to operate at 100 psi and meet the collapse resistance requirements set forth in Specification MIL-H-6000 and flexibility requirements equivalent to Specification MIL-H-5511.

A heat resistant fuel and oil hose equivalent to Specification MIL-H-5511 was developed. This hose is designed to handle MIL-L-7808 type oil at an ambient temperature of 350°F.

Experimental work was conducted to develop a fuel hose equivalent to Specification MIL-H-5511 hose except to meet higher operating pressure requirements. No hose was developed to meet these requirements.

WADC TR 53-373 Sup 2

October 1954

SUBJECT: FUEL CELL SEALANT COMPOUNDS
INVESTIGATOR: Earl H. Sorg, John P. Bowen, Edward M. Fettes, Joseph S. Jorczak
CONTRACT: AF 33(038)-30523
CONTRACTOR: Thiokol Chemical Corporation
ABSTRACT: This work was undertaken by the Thiokol Chemical Corporation to develop integral fuel tank sealant compounds with improved properties.

The hexamethylene terpolymer sealant compound, prepared and submitted to Wright Field at the end of the first year's work on this contract showed better heat resistance coupled with greatly improved low temperature properties than a comparable IP-2 compound. The 0.5 mole % of TCP and 2 mole % of TCF cross-linked pentamethylene formal copolymers showed better heat and aromatic fuel resistance than the 2 mole % of TCP cross-linked copolymers previously submitted.

Compounding studies showed that IP-32 should be more suitable than IP-2 compounds since lower moduli and higher ultimate elongation before and after heat aging and after immersion in aromatic fuel were attained.

Five two-package mix experimental translucent sealant compounds prepared from the liquid polysulfide/epoxy resin system and submitted to Wright Field were tough and resistant to jet fuel but did not pass all of the Air Force specifications. However, the compounds displayed promising potentialities.

The IP-2 base experimental translucent sealant compound submitted to Wright Field for evaluation showed good potentialities as an improved sealant and was more transparent during and after application and cure than an IP-2 base commercial sealant compound.

Many of the materials tested in this investigation were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of the material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

WADC TR 54-62

July 1954

SUBJECT: BUTYL INNER TUBE COMPOUND FOR AIRCRAFT TIRES
INVESTIGATOR: Emmett B. Reinhold
CONTRACT: AF 33(600)-22796
CONTRACTOR: General Tire & Rubber Company
ABSTRACT: This report deals with the development of a butyl rubber compound with requisite physical properties for fabrication into inner tubes for aircraft tires.

A large number of plasticizers of widely divergent chemical characteristics were evaluated, with special emphasis placed on their low temperature properties in butyl rubber and including the technique of using high black,

high plasticizer with a high viscosity elastomer. The effect of zinc oxide content in the formula was determined. Carbon blacks of all commercially available types were compared and the effect of the various blacks on physical properties, including low temperature characteristics were determined. On blacks producing highest tensile values, series of tests were made to determine the loading which produces the maximum physical properties. A comparison of low temperature properties is made of the available commercial butyl rubbers. An extensive investigation of an outstanding low temperature material, Silicone, was carried out.

On plasticizers which produced adequate low temperature flexibility in butyl rubber a study is made on volatility and migration of the plasticizer from cured compound and then low temperature properties determined following migration treatment of cured tensile sheets. In all cases, plasticizers which produced the target low temperature requirements showed poor low temperature properties following migration treatment.

The effect of inorganic acceleration and of the recently recommended processing technique of high temperature mixing was investigated.

WADC TR 54-190

January 1955

SUBJECT: DEVELOPMENT OF HIGH-TEMPERATURE OIL-RESISTANT RUBBER
INVESTIGATOR: William H. Gillen, William J. Mueller, Richard A. Clark,
Randall G. Heiligmann
CONTRACT: AF 33(616)-476
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: Research toward the development of rubber compounds, to be used in connection with a diester-type lubricating oil (Turbo Oil-15) for long term exposure at 350 to 550°F, is described in this report. The evaluation of experimental compounds was confined to one temperature (350°F). At this temperature, the most promising results were obtained with compounds prepared from an acrylate polymer, Hycar 4021, compounded with Silene EF. The best composition of this type fell short of the target requirements only because of about 6 per cent excessive swelling. Another acrylate-type rubber, Acrylon EA-5, shows about equal promise for this application.

Compounds of a butadiene-acrylonitrile copolymer (Hycar 1001) showed promise, except that they cracked badly when aged in Turbo Oil-15 at 350°F. EIC Magnesia was the best reinforcing agent used with this polymer. Variations in the antioxidant and vulcanizing system provided only slight improvement in crack resistance. The chief weakness of this polymer is the vulnerability of its double bonds to oxidation.

The emphasis of future research on this project will be directed toward compounding acrylate-type rubbers, including poly-1,1-dihydro-perfluorobutyl acrylate, and in seeking methods for protecting the double bonds of acrylonitrile-type rubber.

WADC TR 54-190 (Part 2)

April 1955

SUBJECT: DEVELOPMENT OF HIGH TEMPERATURE OIL RESISTANT RUBBER
INVESTIGATOR: William J. Mueller, Louis E. Novy, Richard A. Clark
Randall G. Heiligmann
CONTRACT: AF 33(616)-476
CONTRACTOR: Battelle Memorial Institute

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ABSTRACT: This report describes additional research directed toward the development of rubber compounds that can be used in diester-type lubricating oil (Turbo Oil-15) for long exposures at 350 to 550 F. The evaluation of experimental compounds was confined largely to 350 F. although a few tests were made at 400 F. Studies at Battelle showed that poly-FBA (poly-1,1-dihydroperfluorobutyl acrylate) can be compounded to meet the minimum target requirements of this project. Further work with the acrylate-type rubbers, Hycar 4021 and Acrylon EA-5, showed no substantial gain for this class of rubbers. Hycar 4021 still misses the target requirements only because of a swell that is 6 per cent above the 30 per cent maximum desired. Acrylon EA-5 appears inferior to Hycar 4021 for this application because of its tendency to crack during hot-oil aging tests. This cracking tendency is unusual, inasmuch as the material is a saturated polymer.

Further attempts have been made to overcome the only real short coming of high-nitrile rubbers (such as Hycar 1001) for this application--cracking after hot-oil aging. Although some short-term improvements have been effected by employing thiols and amines as antioxidants, all such gains have been lost by the end of 500 hours' aging. Methods for eliminating a portion of the polymer double bonds by hydrogenation are under consideration. The double bonds or hydrogen atoms on carbons alpha to the double bond are presumed to be focal points for oxidative degradation.

Two aluminum-block heaters for test-tube aging of rubber samples in oil at temperatures up to 500 F and 800 F, respectively, have been designed for WADC under this contract.

In future work on this basic problem, the emphasis will be shifted toward developing rubber compositions for use in synthetic hydraulic oils, such as the silicate esters.

WADC TR 54-213

July 1954

SUBJECT: ELASTOMERIC FLUOROALKYL SILOXANE COPOLYMERS

INVESTIGATOR: Roy T. Clark, Jr.

ABSTRACT: Preparation of fluorine-containing silicone heteropolymers has been accomplished on an experimental scale. Specifically, elastomeric copolymers have been prepared of dimethyl dichlorosilane with bis 3,3,4,4,5,5,5 heptafluoropentyl diethoxysilane, 3,3,4,4,5,5,5 heptafluoropentyl methyl diethoxysilane and bis 3,3,3 trifluoropropyl dimethoxysilane. Copolymerization was realized both from the oil resulting from cohydrolysis of these compounds and by polymerizing a blend of the separately hydrolyzed materials.

These copolymers all exhibited improved resistance to the swelling action by fuels and retained other desirable physical properties to a large degree when compared to methyl silicone rubber prepared

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by the same method. Although these elastomers, like commercial silicone rubber, had rather low tensile strength when in the swelled condition, they had considerably greater stability in diester oil at elevated temperature than either methyl silicone rubber prepared in the same manner or the commercially available polymers; the fluorinated elastomers retain over 50% of their tensile strength after 24 hours in diester fluid at 400°F, whereas the unfluorinated materials dissolve at approximately 350°F. Attempts to prepare fluorine-containing silicone homopolymers have been unsuccessful.

WADC TR 54-318

August 1954

SUBJECT: DEVELOPMENT OF A RUBBER FOR HIGH TEMPERATURE SERVICE IN
CONTACT WITH EXPERIMENTAL HYDRAULIC FLUIDS

INVESTIGATOR: Frederick G. Kitts

ABSTRACT: The proposed operation of hydraulic systems at temperatures in excess of 500°F made necessary the development of a rubber for packings, gaskets, hose, etc., which would withstand newly developed fluids at system temperatures the use of standard packings was proven to be impracticable.

Various commercial and experimental polymers were evaluated at elevated temperatures and ethyl acrylate copolymers were found to have the best inherent resistance to the fluids at high temperatures. Compounding studies were made on two such copolymers with particular emphasis on curing systems and fillers.

A compound was developed which laboratory tests indicate is satisfactory. "O" ring seals have been fabricated of this material and are now awaiting evaluation in actual hydraulic system components and mock-ups. It is anticipated that satisfactory hose can also be fabricated from this compound.

WADC TR 54-407

November 1954

SUBJECT: RESEARCH AND DEVELOPMENT OF NONMETALLIC ACID-RESISTANT HOSE

INVESTIGATOR: Donald C. Dougall, Stephen Casapulla

CONTRACT: AF 33(616)-425

CONTRACTOR: The Connecticut Hard Rubber Company

ABSTRACT: This report describes the development and fabrication of a flexible nonmetallic acid-resistant hose for use on type B-2 servicing semi-trailers. The final product of this research was the fabrication of two fifty-foot lengths of hose, consisting of a polytrifluorochloroethylene helical bellows liner, encased in a synthetic rubber tube and a stainless steel abrasion-resistant cover. The hose is capable of resisting the corrosive effects of red and white fuming nitric acid. It will withstand a pressure of 125 psi, will operate at temperatures from -65° to 160°F, and can be used as a suction hose.

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The development and research were carried out by The Connecticut Hard Rubber Company of New Haven, Connecticut

WADC TR 54-458

December 1954

SUBJECT: DEVELOPMENT OF A RUBBER FOR SERVICE IN CONTACT WITH EXPERIMENTAL HYDRAULIC FLUIDS AT 400°F

INVESTIGATOR: Frederick G. Kitts

ABSTRACT: The operation of hydraulic systems of piloted supersonic aircraft at temperatures in the neighborhood of 400°F will necessitate the replacement of standard AN seals and hose with a more heat and fluid resistant compound. Earlier work had proved Buna N (Butadiene-Acrylonitrile Copolymers) unsatisfactory over the temperature range -65° to 400°F in the silicate ester type fluids so Neoprene WRT was chosen as the best overall compromise of original and aged physical properties.

A compound of Neoprene WRT was developed which was marginally satisfactory after aging 168 hours in MLO 8200 at 400°F in the absence of air. If air were not at least partially excluded the rubber would reach an unsatisfactory condition in the less than 70 hours. It was found that a blend of 85 parts of MLO 8200 and 15 parts of di(2 ethyl hexyl) sebacate with anti-oxidant added to bring the concentration in the blend up to the original concentration in MLO 8200 gave the best balance of aged properties with Neoprene WRT with and without air above the fluid surface.

The compounds of Neoprene WRT are believed to be processable on industrial equipment such as mills, extruders, etc. "O" rings have been fabricated in the Materials Laboratory and are undergoing evaluation testing.

WADC TR 55-26 (Part 1)

March 1955

SUBJECT: RESEARCH ON BORON POLYMERS LITERATURE SURVEY

INVESTIGATORS: William L. Ruigh, Charles E. Erickson

CONTRACT: AF 33(616)2057

CONTRACTOR: Rutgers University

ABSTRACT: A literature search has been made in the field of boron polymers. Polymers derived from boric acid esters are all sensitive to hydrolysis by water as are borazole and its derivatives. Boronamides and N-boro-ureas have been claimed to yield strong fibre and film-forming polymers.

The role of boron compounds in the technology of silicone and other synthetic rubbers is important. The patent literature does not satisfactorily reveal the chemical functions played by boron compounds.

Research has been initiated on the preparation of polymeric boron-amides derived from stabilized boronic acids and bifunctional isocyanates.

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SUBJECT: RESEARCH ON BORON POLYMERS
INVESTIGATOR: W.L. Ruigh, C.E. Erickson, F. Gunderloy, M. Sedlak
CONTRACT: AF 33(616)-2057
CONTRACTOR: Rutgers University

WADC TR 55-57

April 1955

SUBJECT: VULCANIZATION OF RUBBER WITH HIGH-INTENSITY GAMMA RADIATION
INVESTIGATOR: Wallace W. Jackson, Denver Hale
ABSTRACT: Techniques of vulcanizing both natural and synthetic rubber polymers, using high intensity gamma radiation are described.

This process utilizes the gamma radiation from Cobalt 60 or spent uranium reactor fuel elements and is accomplished without heat or chemical vulcanizing agents. Reinforcing agents, anti-oxidants and other additives are still required however, to obtain an optimum balance of physical properties.

After compounding the elastomers in the conventional manner and subjecting them to various dosages of gamma radiation, the resultant physical properties were determined.

Evaluation of this technique shows considerable promise in developing elastomers for specialized uses, such as producing high temperature oil resistant rubber compounds and for improving the compression set of specialized rubber compounds.

This study further emphasizes the practical usefulness of atomic energy in providing the rubber technologist a new experimental variable or tool for determining fundamental properties of elastomeric compounds.

WADC TR 55-117

June 1955

SUBJECT: ELASTOMERIC DITHIOPOLYESTERS
INVESTIGATOR: F.W. Knobloch
ABSTRACT: Elastomers have been prepared from an experimental liquid polymer formulation derived from a condensation product of mercaptoacetic acid and triethylene glycol. Two general types of these elastomers are discussed; one was obtained by reaction of the liquid polymer with organic and inorganic peroxides, the other by reaction of terminal mercapto groups with unsaturated aldehydes.

Selected specimens of each type of elastomer were successfully compounded and vulcanized using conventional rubber processing equipment. The behaviour of these vulcanizates in fuels and synthetic ester base oils has been

studied along with the effects of aging at elevated temperatures. The vulcanized dithiopolyesters showed excellent resistance to the swelling action of 70/30 iso-octane/toluene fuel mixtures. Ester base oils of the MIL-L-7808A type were found to cause shrinkage. Elevated temperatures (350°F) consistently brought about rapid deterioration of both vulcanized and raw polymer stocks.

WADC TR 55-193

June 1955

SUBJECT: FLUORINE-CONTAINING POLYETHERS
INVESTIGATOR: Ogden R. Pierce, Donald D. Smith, Robert M. Murch
CONTRACT: AF 33(616)2417
CONTRACTOR: Dow Corning Corporation
ABSTRACT: Appreciable quantities of the two monomers, $\text{CF}_3\text{CH}-\text{CH}_2\text{O}$ and $\text{CF}_3\text{C}(\text{CH}_3)\text{CH}_2\text{O}$ have been prepared by reactions involving the dehydrohalogenation of the corresponding halohydrins.

Dehydrohalogenation studies were conducted using the following halohydrins: $\text{CClF}_2\text{CH}_2\text{OH}$, $\text{CCl}_2\text{FCH}_2\text{OH}$, $\text{CClF}_2\text{CHOHCCl}_2\text{F}$, $\text{CClF}_2\text{CHOHCClF}_2$, and $\text{CCl}_2\text{FCHOHCCl}_2\text{F}$. The corresponding epoxides were not obtained but rather extensive decomposition of the organic reactants was observed.

An investigation of the hypochlorination of the olefins, $\text{CH}_2=\text{CF}_2$, $\text{CF}_3\text{C}(\text{CH}_3)=\text{CH}_2$ and $\text{C}_3\text{F}_7\text{CH}=\text{CH}_2$, is in progress. The results are inconclusive at this time.

The preparation of a polyglycol formal was accomplished by reaction of a fluorine-containing glycol with dibutyl formal. The material was found to be thermally unstable.

The polymerization of both $\text{CF}_3\text{CHCH}_2\text{O}$ and $\text{CF}_3\text{C}(\text{CH}_3)\text{CH}_2\text{O}$ was accomplished using ferric chloride as a catalyst. In addition, copolymers of these two monomers were prepared in a similar manner.

SANDWICH CONSTRUCTION

WADC TR 52-184 (Suppl. 2)

February 1955

SUBJECT: SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTION FOR AIRCRAFT
INVESTIGATOR: Donald C. Coleman
CONTRACT: D.O. 33(616)53-20
CONTRACTOR: Forest Products Laboratory
ABSTRACT: Developments in the program of research in sandwich aircraft construction conducted by the U.S. Forest Products Laboratory during fiscal year 1954 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Three technical reports issued during the fiscal year are abstracted.

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SUBJECT: DEVELOPMENT OF A HEAT-RESISTANT FOAMED-IN-PLACE LOW-DENSITY SILICONE RESIN CORE MATERIAL

INVESTIGATOR: Donald E. Weyer, James R. Russell, Kenneth R. Hoffman

CONTRACT: AF 33(600)6320

CONTRACTOR: Dow Corning Corporation

ABSTRACT: Methods for producing foamed-in-place sandwich structures from silicone resins have been developed. The most promising method consists of expanding a dry powdered resin containing blowing agent, catalyst and inert filler. The powder melts and expands readily on heating. Core density can be conveniently controlled by adjustments in the expansion temperature. Further density control is possible with small modifications in formulating the powder.

The expansible powder produces a stable, uniform multipore foam with a pore structure predominately spherical and unicellular. None of the materials or by-products are toxic. The foams have low moisture absorption along with excellent electrical properties. They are nonflammable and very resistant to an open flame.

Foamed-in-place sandwich panels have been consistently reproduced. Density variation among the panels was ± 1 lb./cu.ft. Sandwich panels were made with core density ranging from 11 to 24 lbs./cu.ft. At room temperature the compressive strength of these panels varied from 80 to 400 psi. Compressive strength after 1/2 hour exposure to 500°F was from 20 to 100 psi. Specifically, a panel having a density of 18 lbs./cu.ft. had a room temperature compressive strength of 200 psi. After 1/2 hour and 200 hours at 500°F its compressive strength was 50 and 70 psi respectively. Room temperature tensile strength of this panel averaged 60 psi.

Thermal life of the core was over 1000 hours at 600° with no appreciable weight loss or dimensional change. Weight loss of the core after 72 hours at 700°F was less than 6 percent.

WADC TR 54-138

July 1954

SUBJECT: COMPARISONS OF TEST METHODS FOR EVALUATING ADHESIVES FOR BONDING METAL FACINGS TO METAL HONEYCOMB CORES

INVESTIGATOR: H.W. Eickner, Fred Werren

CONTRACT: AF 33(038)51-4326E

CONTRACTOR: Forest Products Laboratory

ABSTRACT: Various shear, tension, and peel tests for determining the quality of adhesive bonds in sandwich panels were compared on matched specimens for sandwich panels of aluminum facings bonded to aluminum honeycomb core with four adhesive processes.

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Flatwise shear and flexural tests gave similar shear values, with failures generally occurring in the core. The tension tests gave good indication of relative strengths of the bonds when stresses were normal to the glue line.

The peel tests included direct peel and drum peel, under both static and impact loading. These tests ranked the peel resistance of bonds in approximately the same order. It appears that there can be standardization on a simple method to rank peel resistance in the same order as do the complicated and wide variety of peel-test methods in use.

Since each type of test, shear, tension, and peel, seemed to evaluate different properties, all should be used for a complete evaluation of adhesives.

TESTS, NONDESTRUCTIVE

WADC TR 54-231 (Part 1)

October 1954

SUBJECT: DEVELOPMENT OF NONDESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS
INVESTIGATOR: J.S. Arnold
CONTRACT: AF 33(616)-2035
CONTRACTOR: Stanford Research Institute
ABSTRACT: Research has been done on the use of sonic methods for the non-destructive evaluation of metal-to-metal adhesive bonds. No aspect of the low frequency behavior (less than 15,000 cps) has been found to be indicative of joint strength. Techniques utilizing high frequency impedance measurements as provided by mechanical driving systems, and steady displacements produced by the application of vacuum cups, have been developed. These techniques show sufficient correlation between bond strength and measurable parameters to justify further investigation, and one or more of them may ultimately provide a satisfactory solution to the non-destructive test problem for adhesive bonds.

WADC TR 54-231 (Part 2)

May 1955

SUBJECT: DEVELOPMENT OF NON-DESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS. PART 2 MECHANICAL INTERFERENCE TECHNIQUE
INVESTIGATOR: J.S. Arnold
CONTRACT: AF 33(616)2035
CONTRACTOR: Stanford Research Institute
ABSTRACT: A mechanical interference technique is described for use in the evaluation of adhesive bonds. This technique uses an optical-mechanical method for measuring small displacements of the skin of a honeycomb sandwich panel as a vacuum-cup induced load is applied. The indication of defects is presented visually in terms of a changing pattern of light and dark "mechanical interference fringes." The technique is a comparatively simple one, but its range of applicability seems to be limited to certain classes of panel defects.

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SUBJECT: DEVELOPMENT OF NONDESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS

INVESTIGATOR: J.S. Arnold

CONTRACT: AF 33(616)2035

CONTRACTOR: Stanford Research Institute

ABSTRACT: An ultrasonic technique is being developed for the non-destructive evaluation of structural adhesive bonds. A ferroelectric transducer is excited by a variable frequency driving current of constant amplitude, and the voltage developed across the transducer is displayed on an oscilloscope as a function of frequency. The voltage-frequency curve conveys information regarding the motional impedance of the system and hence the constraints that limit the vibration of the transducer. These constraints are imposed partly by the mechanical properties of the transducer and partly by the mechanical properties of the load (test specimen) to which it is coupled. The variation in the voltage-frequency curves that occur when the transducer is applied to satisfactory and defective test specimens can usually be correlated with the strength of the adhesive bonds, as indicated by subsequent destructive tests performed on the specimens. Block circuitry, typical oscilloscope patterns, and data on the correlation between destructive and non-destructive tests are presented.

TEXTILES

WADC TR 52-283 (Part 3)

January 1955

SUBJECT: AIR PERMEABILITY OF PARACHUTE CLOTHS PART 3. EFFECT OF LOADING ON ELASTIC PROPERTIES OF PARACHUTE CLOTHS WITHOUT AIR FLOW

INVESTIGATOR: H.W.S. Lavier

CONTRACT: AF 33(038)-15624

CONTRACTOR: Georgia Institute of Technology

ABSTRACT: The effects of biaxial tension loads on fabric samples of nylon, Orlon, and Dacron have been studied. The biaxial-fabric-tension-testing machine, used in these tests, was designed and constructed at Georgia Tech.

It was the purpose of these studies to determine the elastic characteristics of selected nylon, Orlon, and Dacron parachute-type cloth under conditions of no air flow. Also, the effect of high temperatures (+140°F) and low temperatures (-14°F) on elastic properties was determined, using plain-weave samples of nylon, Orlon, and Dacron cloth. The data obtained from these tests were used in conjunction with the low- and high-pressure air-permeability studies reported in AF Technical Reports 52-283, Parts 1, 2, and 4.

Variations in temperature and humidity, in the ranges studied, had a marked effect on the elastic properties of the fabrics. However, as humidity was lowered, the elasticity and the tenacity of the fabrics decreased.

Dacron was found to be somewhat more elastic than Orlon, particularly at the lower temperatures.

The number of picks per inch and the weave pattern (plain, twill, and satin) do not markedly affect the elastic properties.

WADC TR 52-283 (Part 4)

February 1955

SUBJECT: AIR PERMEABILITY OF PARACHUTE CLOTHS
INVESTIGATOR: H. W. S. Lavier
CONTRACT: AF 33(038)-15624
CONTRACTOR: Georgia Institute of Technology
ABSTRACT: The high-pressure air permeability of selected nylon, Orlon, and Dacron parachute-type fabrics was determined using a 16-square-inch sample. The Georgia Tech high-pressure permeometer used in this program permitted testing the fabric samples at pressure differentials across the cloth equivalent to 1500 inches of water. The selected cloths are described in Table I and include experimental cloths woven in the Laboratories of the Georgia Institute of Technology.

Air-permeability data for the selected fabrics are presented here in graphical and tabular form as volumetric flow (cubic feet per minute) and effective porosity versus the static pressure differential across the cloth.

The selected fabrics were chosen to demonstrate the effect on high-pressure air permeability resulting from variation of the number of ends and picks per inch, weave patterns, and material. Also, the effect on high-pressure permeability, due to variation of temperature and absolute humidity, was investigated.

WADC TR 52-283 (Part 5)

April 1955

SUBJECT: AIR PERMEABILITY OF PARACHUTE CLOTHS
INVESTIGATOR: H.W.S. Lavier, W.C. Boteler
CONTRACT: AF 33(038)15624
CONTRACTOR: Georgia Institute of Technology
ABSTRACT: The air permeability evaluations of various nylon cloths produced by different manufacturers to the same MIL specifications are presented here. The effect on air permeability due to various weaving defects was investigated and it was concluded that such normal defects had little or no effect on the cloth air permeability evaluations.

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Variation of the magnitude of the calendering load was studied regarding its effect on air permeability of a cloth. It was found that loads up to fifty tons per inch caused considerable change in the cloth permeability. However, loads above fifty tons per inch caused very little additional change in air permeability over that caused by a load of 50 tons per inch.

Discharge coefficients for various kinds of parachute cloths are also included in this report. These values may prove useful in designing new parachute textiles.

WADC TR 54-49

July 1954

SUBJECT: SYMPOSIUM ON PARACHUTE TEXTILES
INVESTIGATOR: Joyce C. McGrath
ABSTRACT: Technical papers concerning recent developments and future requirements of parachute textile problems were presented at a WADC Symposium on 21 and 22 September 1953. In particular, recent developments in the studies of friction damage, effects of high temperature, air flow through cloth, and design data for use in developing parachute textile materials which will operate at high speeds were discussed.

WADC TR 54-52

November 1954

SUBJECT: EVALUATION OF EXPERIMENTAL WOOL SYNTHETIC BLENDS IN AIR FORCE
18 OZ. BLUE SERGE
INVESTIGATOR: C.A. Willis, C.W. Long
ABSTRACT: A group of twenty-one serge fabrics was evaluated in the laboratory to determine the effects on fabric properties when the fiber and percentage of fiber was varied. The fabrics included the 100% wool standard serge and twenty wool/synthetic blends. The fibers employed in the course of manufacture were wool, Dacron, Orlon, Acrilan, viscose rayon and Dynel. Each synthetic fiber was blended with wool in percentages of 10, 25, 40 and 60.

Comparative tests of the following properties versus wool percentage of the blends were conducted: warp breaking strength, filling breaking strength, warp percent shrinkage, filling percent shrinkage, warp abrasion, filling abrasion, flat abrasion, percent wrinkle recovery, flame time, char rate and wicking time.

Based on laboratory test results of the five synthetic fibers used and the four percentages blended with wool, Dacron, when blended in the fabric appears superior to the other four synthetics in increasing resistance to flat and flex abrasion, and increased breaking strength when used in higher percentages.

In all cases the rate of burning and wicking was increased, and the percent wrinkle recovery slightly decreased, by the addition of synthetic fiber.

Conclusions drawn from the laboratory tests are of interest; however, laboratory testing techniques are not to be considered an absolute measure when evaluating differences between wool and wool/synthetic blends.

WADC TR 54-93

October 1954

SUBJECT: COATED FABRIC FOR USE IN PROTECTIVE CLOTHING
INVESTIGATOR: H. N. Homeyer, Jr., J. Becker
CONTRACT: AF 33(616)-155
CONTRACTOR: The Connecticut Hard Rubber Company
ABSTRACT: Experimental work leading to the development of a chemically resistant coated fabric, suitable for use in protective clothing for personnel handling various fuels and oxidizers used in guided missiles, is described in this report. The fabric consists of a pigmented blend of polyethylene and Vistanex B-80 calendered on Vinyon Fabric #501, primed with a solution (18% solids) of Vistanex B-80 dispersed in toluol. It possesses good resistance to the action of nitric acid and excellent flexibility throughout a temperature range of -67°F to +157°F when tested in accordance with the procedure outlined in Specification MIL-F-4143 (USAF).

Protective clothing in the form of suits, hoods and gloves, fabricated from the coated material, as well as a number of yards of the fabric itself, has been shipped to the Wright Air Development Center. Since the coated fabric which was been developed is superior to suit materials in present use which exhibit only slight resistance to nitric acid, it is concluded that personnel operating in the field of guided missiles can now be provided with clothing which will offer adequate protection against rocket fuels. It is recommended that the coated fabric be used in the fabrication of all clothing designed for the protection of personnel engaged in handling oxidizers and most rocket fuels.

The coated fabric was developed under Contract No. AF 33(616)-155 at The Connecticut Hard Rubber Company, New Haven, Connecticut, during the period from May 22, 1952 to December 31, 1953.

WADC TR 54-117

July 1954

SUBJECT: A STUDY OF THE EFFECT OF TEMPERATURE ON PARACHUTE TEXTILE MATERIALS
INVESTIGATOR: James W. Muse, Jr.
ABSTRACT: The primary purpose of this investigation was to obtain data on the effect of oxygen, nitrogen, and compressed air on nylon and Dacron materials at various temperatures. A secondary purpose was to determine the effect of hot air, applied continuously and in an intermittent manner to parachute textile materials. The test method is described in the text of this report.

A group of standard nylon parachute textile materials comprised of webbings, cord and fabrics, was tested after exposure to various temperature conditions for continuous and intermittent intervals of time. Also tested were Dacron hot stretched threads and an experimental Dacron fabric.

The breaking strength of several nylon textile materials was found to vary with temperature and duration of exposure time. The nylon materials lost considerably more strength after exposure in hot air at 300°F than at 250°F for the same period.

The breaking strength of nylon webbings, treated with resin in accordance with Specification MIL-W-4088B, was affected more severely than the untreated webbings after exposure to the stated conditions.

The breaking strength of the Dacron hot stretched thread showed no appreciable loss in these tests.

The materials tested after exposure in hot air at 250°F for identical continuous and intermittent time intervals show no appreciable differences in breaking strength.

The materials tested after intermittent exposure in hot air at 300°F show more loss in breaking strength than when tested after continuous exposure.

WADC TR 54-199

January 1955

SUBJECT: A STUDY OF THE LAWS OF THE FLOW OF FLUIDS THROUGH FABRICS
INVESTIGATOR: Cecil D. Brown
CONTRACT: AF 33(038)-15624
CONTRACTOR: Georgia Institute of Technology
ABSTRACT: In this study a method is developed of presenting the flow data in a general dimensionless form over a limited range of flow. Under the assumption that the pressure-squared gradient in the flow through a fabric is the arithmetic sum of the viscous ($2\alpha \frac{RT\mu G}{g^c}$) and the inertial contributions ($2\beta \frac{RT}{g^c} G^2$), it is possible to infer the existence of a characteristic length ℓ . The relation between the flow-through-drag coefficient and the Reynolds number based on this characteristic length, $C_f = \frac{2}{N_{Re}} + 2$, is common to all fabrics. The parameters α and β and the characteristic length can be obtained from only two flow measurements.

The parameters α and β may be estimated from physical measurements of the cloth, thus permitting a rough prediction of the permeability of the fabric.

WADC TR 54-374

June 1955

SUBJECT: FIBRE FABRICS FOR INSULATION
INVESTIGATOR: C. W. Long

WADC TR 53-373 Sup 2

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ABSTRACT: This is a report on pile fabrics made from synthetic fibers, cotton, wool, and numerous blends thereof. There were three techniques employed in the construction of the pile fabrics developed under this project: (1) woven cut pile fabrics by Goodall Sanford, Inc., (2) inserted pile, knitted fabrics by George W. Borg Corporation, and (3) napped and/or brushed pile, knitted fabrics by Princeton Knitting Mills, Inc. All the samples developed were compared to the standard wool pile fabric made according to requirements of AF Specification MIL-C-5563. Each sample was tested for warmth and compression characteristics to determine the effect, if any, of varying thicknesses, blends, and constructions. It was observed that the type fiber has little effect on the warmth of a pile fabric; however, Orlon, Dacron, and Dynel consistently appear slightly better. Results show that possibly a double thickness of a relatively thin pile fabric should deserve consideration. Also included in this report are the results of a study on the mathematical relationship between the warmth of a fabric and the physical properties of the fabric.

WADC TR 54-468

March 1955

SUBJECT: STUDY OF THE CONTROL OF PERMEABILITY AT HIGH AND LOW DIFFERENTIAL PRESSURES
INVESTIGATORS: Hamilton J. Bickford, Donald K. Kuehl, Thomas L. Rusk, Jr.
CONTRACT: AF 33(600)26109
CONTRACTOR: Cheney Bros.
ABSTRACT: Twenty-four differently constructed samples of nylon cloth in the desired weight range were woven, finished and tested.

A special mathematical study of the relationship between air permeability at 1/2 inch of water pressure differential and at higher pressure differentials was made. This discloses that a linear relationship in these values exists when plotted on full logarithmic graph paper.

The ability of the cloth manufacturer to vary the high pressure differential permeability, while retaining fixed low pressure permeability ranges is indicated to be a practical one within limits.

A total of 1000 yards of additional cloth duplicating two of the twenty-four constructions as selected by the Air Force was supplied for use in further evaluation of the material by the Parachute Laboratory.

WADC TR 55-3

May 1955

SUBJECT: DEVELOPMENT OF LOW ELONGATION, HIGH STRENGTH WEBBING FOR USE IN SAFETY HARNESS APPLICATION
INVESTIGATOR: Russell J. Neff
CONTRACT: AF 33(616)2361
CONTRACTOR: Phoenix Trimming Co.

WADC TR 53-373 Sup 2

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ABSTRACT: Four types of Dacron webbing, developed by the Phoenix Trimming Company, were subjected to a series of tests to determine whether they were acceptable for use in aircraft safety harnesses. Samples of these webbings were made from three different types of Dacron, with the final delivered webbing being manufactured from 1100-250-0-560 Dacron yarn.

TRANSPARENT MATERIALS

WADC TR 54-57

July 1954

SUBJECT: DEVELOPMENT OF HEAT-RESISTANT INTERLAYER MATERIALS FOR LAMINATED PLASTIC AND LAMINATED GLASS
INVESTIGATOR: Robert P. Cox, Luther L. Yaeger, Ralph W. Buetow, Robert J. Roth
CONTRACT: AF 33(600)-22723
CONTRACTOR: Bjorksten Research Labs, Inc.
ABSTRACT: The following materials were considered for use as heat-resistant interlayer materials for laminated glass and laminated plastic:

- Polyamides (Nylons)
- Polyesters
- Cellulose esters
- Fluorinated resins of the Exon type
- Plasticized Epon resins
- Mylar
- Silicone-acrylate copolymers

The optimum interlayer material was a copolymer of 99 parts of ethyl acrylate and one part of vinyl polysiloxane (Linde X-31 resin). It was prepared as a casting syrup by the photopolymerization of ethyl acrylate to a low molecular weight prepolymer and subsequent mixing with vinyl polysiloxane. Laminates were prepared by curing the interlayer casting resin between two outlayer sheets. Such laminates were prepared with glass, polyester, Plexiglas 55, Polymer K (Rohm and Haas Co.) and Gafite (General Aniline and Film Corp., polymethyl-alpha-chloro-acrylate), as surface sheet materials. These laminates exhibited slight discoloration after six hours at 350°F. and possessed satisfactory projectile and falling ball impact strengths at 0°F.

WADC TR 54-128 (Part 1)

November 1954

SUBJECT: EVALUATION OF MATERIALS FOR GUNSIGHTING DOMES
INVESTIGATOR: John L. Folk, Kenneth R. Iler
CONTRACT: AF 33(616)-106
CONTRACTOR: Goodyear Aircraft Corporation
ABSTRACT: This report covers an evaluation of three laminated transparent acrylic materials for use in the fabrication of aircraft gunsighting domes. Regular grade (BMS 8-4), heat resistant (MIL-P7524), and modified (5105XP) acrylic laminates, all with standard polyvinyl butyral interlayer, were tested. Two laminators supplied the 5105XP test laminates.

WADC TR 53-373 Sup 2

The various testing procedures compared the laminates with respect to (1) ultimate tensile strength at -65°F, room temperature, and 160°F, (2) time required for crazing and failure under long time tensile loads of 750, 1000, 2000, and 4000 psi, and (3) resistance to natural Florida and accelerated weathering. The tests also identified suitable cements for bonding extruded rubber edge attachments of each test material and evaluated the heat stability of the interlayer material.

Of the materials evaluated, laminated 5105XP appears to be the most desirable dome material. Its ultimate tensile strength (5700 psi at 160°F, 10,600 psi at room temperature, and 19,500 psi at -65°F) and its resistance to weathering elements were found to be superior. The long time tensile tests, incomplete at this writing, have indicated to date that the 5105XP laminate will support normal service loads for a longer time than the other two test materials. (Completed data for the long time tensile tests will be submitted later in Part II of this report.)

All of the cementing systems tested would comply with the requirements of normal service specifications, even after weathering. However, a prebodied acrylic monomer type cement, such as GAC Code C-201A, was indicated to be the most satisfactory.

The heat stability (tendency to bubble at normal acrylic forming temperatures) of polyvinyl butyral was decreased with increased moisture content. The heating time and moisture content required to produce bubbling appeared to be independent of the type of laminate or the source, but the location and size of the bubbles differed in the materials obtained from the two laminators. Except for this peculiarity, the 5105XP materials showed little difference in the tested physical properties.

WADC TR 54-207 (Part 1)

October 1954

SUBJECT: DEVELOPMENT OF SILICONE OR FLUOROCARBON INTERLAYER MATERIALS FOR LAMINATED GLASS AND LAMINATED PLASTIC
INVESTIGATOR: Keith E. Polmanteer, John W. Gretzmeyer, John W. Erwin
CONTRACT: AF 33(600)-23081
CONTRACTOR: Dow Corning Corporation
ABSTRACT: The development of a transparent interlayer material for laminated glass possessing thermal stability within the range of 400° to 500°F was the object of this research by the Dow Corning Corporation during the period of 15 April 1953 to 15 April 1954. Nine polysiloxane materials were introduced and evaluated for suitability as interlayers. A Type J interlayer was found which possessed tensile strength of 400 to 800 psi, 80 to 85% transmission, 4 to 10% haze and thermal stability of several hours at 450°F.

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This interlayer lends itself well to commercial fabrication techniques. The optical properties may require some improvement before this material will be completely satisfactory for aircraft glazing.

WADC TR 54-429

October 1954

SUBJECT: ELEVATED-AND ROOM-TEMPERATURE PROPERTIES OF POLYMER K
TRANSPARENT PLASTIC SHEET MATERIAL
INVESTIGATOR: John VanEcho, Gale R. Remely, Ward F. Simmons
CONTRACT: AF 33(038)-10818
CONTRACTOR: Battelle Memorial Institute
ABSTRACT: Creep and creep-rupture, short-time tensile, and deterioration data were obtained on Polymer K, a transparent, thermosetting acrylic sheet material. Tests were made at 80, 160, 200, and 250 F. A comparison of properties is made with several other transparent plastics.

The room-temperature creep and creep-rupture strength of this material is quite similar to that of 5105XP, and Plexiglas II. The creep and creep-rupture strengths at 160 and 200 F are quite superior to any other transparent tested under this program. The short-time tensile strength of Polymer K is inferior to 5105XP and Selectron 44 at room temperature but considerably greater at the higher temperatures.

The deterioration losses of Polymer K are, generally, lower than those for 5105XP and Selectron 44. The test material showed a maximum of 0.7 per cent weight loss after the 1000-hour exposure at 200 F.

WADC TR 55-24

June 1955

SUBJECT: DETERMINATION OF THERMAL SHOCK CHARACTERISTICS OF GLASS
INVESTIGATOR: David Horwitz
CONTRACT: AF 33(616)2240
CONTRACTOR: Armour Research Foundation
ABSTRACT: This is a study of the resistance to thermal shock of glass having different coefficients of linear thermal expansion and different tempers and subjected to shock applied to a single surface. The program was initiated in order to enable the selection of a suitable glass for windshields in supersonic aircraft.

Apparatus was designed to produce a timed sudden shock to a glass specimen at elevated temperatures, simulating skin temperatures which may develop in supersonic aircraft due to aerodynamic heating.

Instrumentation was developed to record "surface" temperature of the specimen independent of the conditions of the attacking fluid.

Time-temperature change curves were drawn, and the shock rates established for the various glasses were used as a means of comparison.

WADC TR 53-373 Sup 2

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SUBJECT: SILICONE INTERLAYER MATERIAL PROGRAM

INVESTIGATOR: Keith E. Polmanteer, Francis J. Campbell, Jack Fenner

CONTRACT: AF 33(600)27185

CONTRACTOR: Dow Corning Corporation

ABSTRACT: The further development of a silicone thermally stable interlayer, originally introduced under AF Contract 33(600)-23081, was the object of this research by the Dow Corning Corporation during an eight and one-half month period ending in January, 1955. The optical properties, tensile strength and thermal stability of this silicone interlayer material were substantially improved in this work. This improved interlayer was identified as Type JA. It possessed 1.8-3.0% haze, 86.5%-87.5% transmittance, 750-1200 psi tensile strength, and thermal stability at 400°F and above for over 100 hours.

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ALLOYS, NONFERROUS, MAGNESIUM

WADC TR 53-405

March 1953

SUBJECT: WADC MAGNESIUM SYMPOSIUM-HELD ON 24 FEBRUARY 1953
INVESTIGATOR: Henry A. Johnson

CRITERIA DESIGN

WADC TR 52-89 (Part 1)

August 1952

SUBJECT: PLASTIC BEHAVIOR OF ENGINEERING MATERIALS PART 1. AXIAL
TENSION AND BENDING INTERACTION CURVES FOR MEMBERS LOADED
INELASTICALLY
INVESTIGATOR: D. O. Brush, O. M. Sidebottom, J. O. Smith
CONTRACT: AF 33(038)-15677
CONTRACTOR: University of Illinois

WADC TR 52-251 (Part 1)

September 1952

SUBJECT: INVESTIGATION OF COMPRESSIVE-CREEP PROPERTIES OF ALUMINUM
COLUMNS AT ELEVATED TEMPERATURES
INVESTIGATOR: R. L. Carlson, A. D. Schwabe
CONTRACT: AF 33(038)-9542
CONTRACTOR: Battelle Memorial Institute

WADC TR 53-24 (Part 1)

May 1954

SUBJECT: INTERMITTENT STRESSING AND HEATING TESTS OF AIRCRAFT
STRUCTURAL METALS
INVESTIGATOR: G. J. Guarnieri
CONTRACT: AF 33(038)-10958
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.

JOINING

WADC TR 53-231

December 1953

SUBJECT: RECRYSTALLIZATION WELDING OF ALUMINUM AIRCRAFT ALLOYS
INVESTIGATOR: Nickolas A. DeCecce, John M. Parks
CONTRACT: AF 18(600)-92
CONTRACTOR: Armour Research Foundation

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PETROLEUM PRODUCTS, LUBRICANTS

WADC TR 52-22

September 1952

SUBJECT: LOW TEMPERATURE LUBRICATING OIL ADDITIVES
INVESTIGATOR: G. Gavlin, S.P. Jones, Jr., E.A. Swire
CONTRACT: AF 33(038)1644
CONTRACTOR: Armour Research Foundation

WADC TR 53-426 (Part 1)

April 1954

SUBJECT: ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS
AND RELATED MATERIALS
INVESTIGATOR: Henry Gilman, Robert K. Ingham, Richard D. Gorsich
CONTRACT: AF 33(616)94
CONTRACTOR: Iowa State College

PLASTICS, STRUCTURAL

WADC TR 52-24

July 1952

SUBJECT: DEVELOPMENTS CONCERNING AIRCRAFT GLASS FIBER PLASTIC
LAMINATES
INVESTIGATOR: Gail A. Clark

WADC TR 53-185

February 1954

SUBJECT: A STUDY OF THE RAIN EROSION OF PLASTICS AND METALS
INVESTIGATOR: Roy R. Lapp, Raymond H. Stutzman, Norman E. Wahl
CONTRACT: AF 33(600)-6469
CONTRACTOR: Cornell Aeronautical Laboratory Inc.

TEXTILES

WADC TR 54-39

June 1954

SUBJECT: AN EMPIRICAL REPORT ON THE EFFECT OF POLYVINYL BUTYRAL ON
CARGO-PARACHUTE SHROUD LINES
INVESTIGATOR: Robert L. Phipps
CONTRACT: AF 33(600)-23556
CONTRACTOR: Thomas Taylor & Sons, Inc.

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